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# INVESTIGATION OF THE FEASIBILITY OF STERILE ASSEMBLY OF SILVER-ZINC BATTERIES

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MARTIN MARIETTA CORPORATION  
Denver, Colorado

for

Langley Research Center  
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

INVESTIGATION OF THE FEASIBILITY  
OF STERILE ASSEMBLY  
OF SILVER-ZINC BATTERIES

By Arnold A. Rothstein, Evan R. Jones,  
and Roger D. Knight

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## FOREWORD

This report is submitted in accordance with Part IIIC of Contract NAS1-7656.

# CONTENTS

	Page
FOREWORD . . . . .	ii
CONTENTS . . . . .	iii
LIST OF ILLUSTRATIONS . . . . .	v
LIST OF TABLES . . . . .	vi
SUMMARY . . . . .	1
INTRODUCTION . . . . .	2
STERILIZATION COMPATIBILITY . . . . .	5
Dry Heat Sterilization Compatibility . . . . .	6
Autoclave Sterilization Compatibility . . . . .	8
Liquid Component Sterilization Compatibility . . . . .	9
Assembly Autoclave Sterilization . . . . .	9
Conclusions . . . . .	12
ASSEMBLY OPERATIONS . . . . .	13
Standard Cells . . . . .	15
Control Cells . . . . .	16
Sterile Assembly . . . . .	17
Conclusions . . . . .	17
ELECTRICAL PERFORMANCE . . . . .	18
Technical Approach . . . . .	18
Standard Cell Performance . . . . .	26
Control Cell Performance . . . . .	31
Sterile Cell Performance . . . . .	35
Cell Performance Comparison . . . . .	46
Conclusions . . . . .	50
BIOLOGICAL ASPECTS . . . . .	51
Preassembly Sterilization Verification . . . . .	52
Assembly Sequence Microbiological Monitoring and Control . . . . .	56
Post-Electrical Test Microbiological Assay . . . . .	59
Conclusions . . . . .	64
PACKAGING CONCEPTS . . . . .	65
Hermetically Sealed Stainless Steel Case . . . . .	66
Thermal Barrier Package . . . . .	67
Heat Exchanger Package . . . . .	68
Shipping Container Service Chamber . . . . .	68
Split Seam Package . . . . .	70
Flap-Type Sterile Seam . . . . .	72
CONCLUSIONS AND RECOMMENDATIONS . . . . .	74
Conclusions . . . . .	74
Recommendations . . . . .	76
APPENDIX A -- EQUIPMENT DESCRIPTION . . . . .	77
Environmental Control Equipment . . . . .	77
Test Equipment . . . . .	80



	Page
APPENDIX B -- STERILIZATION PROCEDURES . . . . .	81
Component Sterilization Tests . . . . .	81
Cellophane Separator Sterilization Tests at 135 and 125°C . . . . .	81
APPENDIX C -- STERILE ASSEMBLY OPERATIONS . . . . .	82
Cell Assembly Equipment and Materials . . . . .	82
Cell Components . . . . .	85
Isolator Decontamination . . . . .	85
Dry Heat Sterilization of Cell Components . . . . .	85
Moist Heat Sterilization Cycle . . . . .	87
Equipment and Material Placement in Isolators . . . . .	89
Isolator Environment . . . . .	89
Step-by-Step Cell Assembly Operation . . . . .	90
APPENDIX D -- CHARGE/DISCHARGE TEST PROCEDURE . . . . .	98
Formation Cycle . . . . .	98
Operational Cycle . . . . .	101
APPENDIX E -- MICROBIOLOGICAL CONTROL AND ASSAY PROCEDURES . . . . .	102
Media Preparation . . . . .	102
Sterilization Process Evaluation . . . . .	102
Microbiological Assay Procedures . . . . .	103
REFERENCES . . . . .	107

# LIST OF ILLUSTRATIONS

Figure		Page
1	Average Charge Curve, ESB S-25, 2-A Charge Rate . .	10
2	Average Discharge Curve, ESB S-25, 8-A Discharge Rate . . . . .	11
3	Cell Assembly and Test Sequence . . . . .	14
4	Average Charge Curves, 2-A Rate . . . . .	20
5	Letdown Discharge Performance Curves, 4-A Rate . .	24
6	Standard Cell Discharge Curves, 10-A Rate . . . . .	28
7	Control Cell Discharge Curves, 10-A Rate . . . . .	32
8	Sterile Cell Discharge Curves, 10-A Rate (135°C Cellophane Cells) . . . . .	36
9	Sterile Cell Discharge Curves, 10-A Rate (125°C Cellophane Activated Cells) . . . . .	40
10	Sterile Cell Discharge Curves, 10-A Rate (125°C Cellophane Stored Cells) . . . . .	44
11	Discharge Minimum and Maximum Values, Cycle 5 . . .	47
12	Average Discharge Comparison, Cycle 5 . . . . .	49
13	Isolator Airborne Contamination Monitoring System .	57
14	Hermetically Sealed Stainless Steel Battery Case Concept . . . . .	66
15	Thermal Barrier Ship and Test Package Concepts . .	67
16	Heat Exchanger Package Concept . . . . .	68
17	Shipping Container Service Chamber Concept . . . . .	69
18	Split Seam Insertion Technique . . . . .	70
19	Split Seam Packaging Concept . . . . .	71
20	Flap-Type Sterile Seam Concept . . . . .	73
A1	Stainless Steel Sterilization Oven . . . . .	78
A2	Isolator Setup . . . . .	79
C1	Cell Assembly Equipment and Materials . . . . .	83
C2	Electrical Checkout . . . . .	85
C3	Cell Components . . . . .	86
C4	Wrapping Positive Plate . . . . .	91
C5	Positive Plate U-Fold . . . . .	92
C6	Inserting Negative Plate . . . . .	93
C7	Soldering . . . . .	95
C8	Filling Cell . . . . .	96
D1	Charging Circuit Diagram . . . . .	99
D2	Discharging Circuit Diagram . . . . .	100
E1	Bioassay of Sterile Assembled Cells . . . . .	105

# LIST OF TABLES

Table		Page
1	Cell Assembly Categories . . . . .	3
2	Effects of 135°C Dry Heat Sterilization on Cell Components . . . . .	6
3	Summary of Tensile Data . . . . .	7
4	Analysis of Variance of Tensile Data . . . . .	8
5	Nylon Cell Case Sealants . . . . .	16
6	Standard Cell Group Charge and Discharge Ampere- Hours . . . . .	30
7	Control Cell Group Charge and Discharge Ampere- Hours . . . . .	34
8	Sterile (135°C Cellophane) Cell Group Charge and Discharge Ampere-Hours . . . . .	38
9	Sterile (125°C Cellophane) Cell Group Charge and Discharge Ampere-Hours . . . . .	42
10	Cell Group Average Data Comparison for Cycle 5 .	48
11	Hardware for Sterile Cell Assembly . . . . .	53
12	Sterilization of Parts for Construction of 12 Sterile Cells (First Set) . . . . .	54
13	Sterilization of Parts for Construction of 12 Sterile Cells (Second Set) . . . . .	55
14	Biological Assay of Sterile Assembled Cells, Battery 1 . . . . .	60
15	Biological Assay of Sterile Assembled Cells, Battery 2 . . . . .	61
16	Biological Assay of Sterile Assembled Cells, Battery 3 . . . . .	62

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OF SILVER-ZINC BATTERIES

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SUMMARY

A program was conducted to ascertain the feasibility of sterile assembly of silver-zinc batteries. A logical plan was followed encompassing (1) sterilization compatibility of the cell components and materials under flight acceptance dry heat sterilization (125°C for 60 hr) and autoclave conditions; (2) assembly of 12 "standard" cells under normal manufacturing conditions, 12 "control" cells from sterilized components using normal manufacturing conditions and 24 "sterile" cells from sterilized components under sterile isolator conditions; (3) electrical performance tests of each type assembly with comparison of charge/discharge data to determine degradation effects; (4) associated biological controls and assays during sterile assembly and post-electrical testing operations; and (5) development of a variety of packaging concepts to maintain sterility in the factory-through-launch sequence.

Component sterilization tests revealed shrinkage of the nylon cell jar and cover and shrinkage/discoloration of the cellophane separator materials. Additional experimentation showed that these effects could be overcome by exposure to high relative humidity after sterilization. Tensile tests revealed no change in properties at a 95% confidence level.

Control and sterile batteries exhibited slight electrical performance degradation. This effect appears to be appreciably less than literature-reported capacity losses from post-assembly sterilization of silver-zinc batteries. No evidence of contamination was detected during the sterile assembly and electrical performance operations. Four positive results were noted in assays of battery components after electrical performance tests that are believed to stem from assay introduced contamination. Six packaging concepts evolved, with two having particular promise and warranting development.

The study results substantiate the feasibility of sterile assembly of silver-zinc batteries as a backup to other investigations to develop sterilizable silver-zinc batteries.

## INTRODUCTION

NASA requirements for sterilization of interplanetary unmanned missions stipulate a terminal heat sterilization cycle equivalent in biological kill effects to exposure at 125°C for 24.5 hr (ref. 1). These conditions introduce potential problems involving repair after the terminal sterilization treatment, and degradation of heat-sensitive hardware by exposure (ref. 2).

To solve the first problem, extensive investigations have been conducted on sterile repair/insertion concepts. The assembly sterilizer approach, developed under the aegis of the Langley Research Center (refs. 3, 4, 5) comprises disassembly and repair in a large sterile chamber. Investigations by the Martin Marietta Corporation under NASA contracts (refs. 6, 7, 8) are based on an insertion technique permitting sterile operations through port openings in the spacecraft. These methods are mutually complementary, and use of one or the other will meet all needs for repair at the launch site. Necessarily, both approaches require the availability of sterile replacement hardware as part of their operations.

Extensive investigations have been sponsored by NASA and various universities and industrial organizations to minimize the second problem by establishing a spectrum of parts, materials, components, and assemblies capable of exposure to sterilization conditions without degradation. Silver-zinc batteries pose a basic problem as a heat-sensitive item exhibiting appreciable degradation in electrical performance capacity characteristics after exposure to the terminal sterilization conditions.

To complement and fill known voids in the existing programs, Martin Marietta Corporation proposed this study under the direction of the Langley Research Center. The program objective was to determine the feasibility of sterile assembly and to develop packaging concepts to maintain sterility during the factory-through-launch sequence. Silver-zinc batteries were selected as the assembly item to concurrently provide a backup technique if other investigations to develop sterilizable batteries were unsuccessful.

The five major phases of this investigation were as follows:

- 1) Component sterilization - This phase was conducted before the start of the assembly operations to establish suitable sterilization conditions for the individual elements of the cells;
- 2) Assembly of test cells - This phase comprised assembly of three categories of cells listed in table 1;

TABLE 1.- CELL ASSEMBLY CATEGORIES

Nomenclature category	Components	Assembly operations
Standard	Nonsterile	Nonsterile
Control	Sterilized	Nonsterile
Sterile	Sterilized	Sterile isolator

- 3) Electrical performance - This phase constituted electrical testing of each category of assembled cells to determine performance characteristics;
- 4) Biological controls and assays - These activities were conducted during the sterile assembly operations, throughout the electrical testing inside the sterile isolators, and as a post mortem analysis on disassembled battery components;
- 5) Packaging concepts - This corollary work developed battery packaging concepts compatible with the Langley Assembly Sterilizer and Martin Marietta sterile-insertion techniques as well as program oriented constraints.

Each of these phases is covered as separate entities in subsequent sections of this report. Detailed procedural information has been incorporated in appendixes to permit ease of reading and reference. Recognizing the wide variety of interest in this work, we have tried to make each section self-sustaining even though some small redundancy is introduced. Materials personnel will find the STERILIZATION COMPATIBILITY section of interest; battery production personnel can extract valuable information from the ASSEMBLY OPERATIONS section and the appendixes; performance characteristics are provided in the ELECTRICAL PERFORMANCE section for battery and space power personnel; etc.

Conclusions and recommendations are provided. Appendix A presents a description of equipment

Martin Marietta Corporation wishes to express our sincerest thanks and appreciation to Jack Zanks, Jim Bene, and John L. Patterson of the Langley Research Center. In our several technical review meetings, these gentlemen, as a team, directed and coordinated the work to yield the requisite blend of battery technology, sterilization operations, and microbiological techniques.

Under the Program Management of Arnold A. Rothstein, Evan R. Jones provided technical direction that assured conformance to all scheduled milestones and coordination of all program activities. Roger D. Knight directed the biological program, and John Leuthard established the basic electrical performance test program. Horace Clair, Eldon Constable, and Al Bowker performed the day-to-day laboratory operations and the battery assembly operations. Special acknowledgment is given to John H. Martin and Charles W. Brooke of the Space Power Laboratory, whose consultation and assistance enhanced the professional stature of our work.

## STERILIZATION COMPATIBILITY

Experimental data on sterilization of completely assembled, potassium hydroxide (KOH) filled, silver-zinc batteries has shown material loss of electrical capacity after dry heat sterilization. This change in performance characteristics is believed to stem from the corrosive effects of the heated concentrated alkali and the susceptibility of some battery components (such as separators) to high-temperature degradation.

Assuming successful evolution of the assembly sterilizer concept and the sterile-insertion concept, sterile batteries could be placed in the spacecraft after the terminal heat sterilization operation. This condition would eliminate heat exposure of the batteries during the launch site operations, but leaves the question of how to obtain sterile batteries. One method offering considerable potential is sterile assembly in an isolator using battery components that have been sterilized in a passclave before introduction into the isolator. This approach tends to eliminate the previously cited problems of sterilizing an assembled battery because:

- 1) The potassium hydroxide could be introduced and sterilized in a separate container. Assembly would then be conducted with the cooled alkali and, hence, eliminate the effects caused by the heated corrosive environment;
- 2) Greater flexibility would be available for the sterilization of battery components to eliminate or minimize degradation. These alternatives in the passclave operation could comprise longer time/lower temperature dry heat exposures or autoclave treatment.

The initial phase of this study was therefore devoted to determining susceptibility to degradation of battery elements under dry heat and autoclave conditions. Biological assays were also conducted as a control aspect on the sterilization treatment and to provide preliminary checkout information on equipment and sterilization procedures in anticipation of the subsequent sterile assembly operations. Results of this work are discussed in this section.



### Dry Heat Sterilization Compatibility

A temperature of 135°C is presently stipulated as the qualification temperature for parts and materials (ref 9). This temperature was therefore used as the initial exposure to restrict deviations from standard practices to those components that showed detrimental effects under the 135°C conditions. All components (except the 40% KOH electrolyte and the ethyl alcohol used in the cell case sealant) were subjected to this dry heat sterilization cycle following the procedure shown in Appendix B. Components were measured before and after the treatment as shown in table 2.

TABLE 2.- EFFECTS OF 135°C DRY HEAT STERILIZATION ON CELL COMPONENTS

Component	Dimension before cycle		Dimension after cycle	
Positive plate	Height	2.50 in.	No change	
	Width	1.18 in.		
	Thickness	.024 in.		
Negative plate	Height	2.50 in.	No change	
	Width	1.81 in.		
	Thickness	.033 in.		
Nylon taffeta	Width	3.37 in.	No change	
	Length	4.25 in.		
Nylon cover	Width	1.580 in.	Width	1.560 in.
	Length	1.95 in.	Length	1.945 in.
	Thickness	.120 in.	Thickness	.118 in.
	Weight	7.9168 g	Weight	7.8609 g
Nylon jar (inside dim)	Width	1.891 in.	Width	1.858 in.
	Diameter	1.583 in.	Diameter	1.561 in.
	Height	3.575 in.	Height	3.511 in.
	Thickness	.070 in.	Thickness	.069 in.
	Weight	40.6842 g	Weight	40.2134 g
Terminal hardware	No measurements taken		No visible change	
Cellophane	19.1x5.87 in.		18.725x5.75 in. darkened color, brittleness	

Significant changes were observed with the nylon case and cover and with the cellophane separators. These changes are discussed below.

Nylon case and cover. - The dimensional shrinkage in these components was sufficient to prevent assembly of the plate packets into the case. Because this shrinkage could stem from loss of water in the dry heat cycle, a small experimental program was instituted. The case and lid were placed in an autoclave (steam sterilizer) and subjected to 121°C at 20 psi steam for 20 minutes. The cases were then removed and checked dimensionally. Under these conditions, the case and the lid had returned to within the original tolerances and were then suitable for assembly. Although suitable for assembly, it should be recognized that the original loss may have included volatile materials other than water, and dimensional stability may change with time.

Cellophane separator materials. - Darkening of the samples, embrittlement, and dimensional shrinkage were of sufficient magnitude to preclude use of the material. To rectify these degradation effects, two experimental programs were conducted.

Dry heat sterilization at 125°C: Four new cellophane sheets were cycled at 125°C for 60 hr following the procedure used for the 135°C samples. Although darkening and embrittlement were slightly less, dimensional shrinkage was comparable to the earlier samples and therefore unsatisfactory for assembly use.

Autoclave vs dry heat with moisture renewal: In view of the favorable results from humidification of the nylon components, a similar study warranted investigation with the cellophane separators. Because discoloration and embrittlement are unsuitable characteristics for correlation with degraded physical properties, tensile strength measurements were introduced as a criterion. Test samples (1x5 in.) were cut from a single sheet of 193 PUD-O cellophane with the long (5 in.) dimension perpendicular to the machine direction of the sheet. Four samples (autoclave) were exposed to autoclave treatment at 121°C and 21 psi for 20 minutes; four samples (dry heat) were exposed to dry heat sterilization at 125°C for 60 hr; and four samples (control) were untreated.

Before tensile testing, all samples were allowed to remain in an ambient atmosphere for 48 hr to permit possible recuperation of lost moisture. Three samples from each group were tested on an Instron Model Load Tester; results are given in table 3.

TABLE 3.- SUMMARY OF TENSILE DATA

Sample	Tensile load, lb		
	Control	Autoclave	Dry heat
1	27	27.5	27.0
2	28	25.0	27.0
3	27	25.0	22.0

An analysis of variance was conducted on the tensile data. The null hypothesis used was that the data stemmed from a homogeneous normal distribution. At a confidence level of 0.95, the test of significance showed an appreciably lower F ratio for the test data than the standard F value. Accordingly, from a statistical standpoint, no difference exists, and cellophane sterilized by either procedure is acceptable from a tensile property standpoint. It was further concluded that electrical performance tests on cells constructed with the sterilized cellophane would be required to ascertain degradation phenomena. Table 4 shows the analysis of variance values.

TABLE 4.- ANALYSIS OF VARIANCE OF TENSILE DATA

	$\Sigma$ of square	df	Mean square	F ratio
Category mean	6.49	2	$s_m^2 = 3.27$	$F = \frac{3.27}{3.74} = .874$
Within	21.51	6	$s_p^2 = 3.74$	$F_{(.95)} = 5.14$
Total	28.00	8		

#### Autoclave Sterilization Compatibility

All materials and components used in assembly of batteries except the liquid materials (discussed below) were subjected to additional tests to establish compatibility with autoclave techniques for sterilization. These tests were performed principally for information purposes because dry heat was stipulated as the preferred sterilization method.

Experimental procedure. - Appendix C describes the autoclave procedure that can be summarized as exposure to steam at 121°C and 21 psi for 20 minutes followed by stabilization at room ambient conditions for 24 hr. Test items were then inspected for dimensional and visual evidence of degradation.

Test results. - Except for the cellophane samples, no apparent physical changes were noted. Unsupported films of cellophane showed dimensional shrinkage of about 5%. As described earlier, tensile tests showed no significant change in properties in the untreated, autoclaved, and dry heat with humidity regeneration cellophane samples. Dry heat was selected for the subsequent assembly operations because of its priority stipulation in the contract provisions.

### Liquid Component Sterilization Compatibility

Potassium hydroxide is used as the cell electrolyte and ethyl alcohol is needed to prepare the case sealant. Separate sterilization tests were therefore required for these chemicals to assure sterility in their containers when subjected to dry heat sterilization in the passclave.

Experimental procedure. - For this experiment, 250 ml of each liquid material was placed in two 304 stainless steel Hoke cylinders. A spore strip was added to each cylinder, and the cylinders were sealed with a screw plug wrapped in Teflon tape. The sealed cylinders were then placed in the autoclave chamber and heated at 125°C for 60 hr. After heating, the cylinders were cooled, removed from the chamber, and opened. The spore strip from each cylinder was removed and incubated in 50 ml of neutral ( $\approx$ pH 7) TSB.

Test data. - No growth was noted on the spore strips after sterilization, which indicated that the procedure was satisfactory for transfer of these liquids.

### Assembly Autoclave Sterilization

As an adjunct to this study, it appeared desirable to identify the highest level of assembly that could withstand autoclave sterilization. A small investigation was therefore conducted.

Experimental procedure. - A standard ESB S-25 cell was procured from the ESB production line at the point of sealing the lid to the case. The cell was prepared for autoclaving by pulling the cell pack and lid up approximately 1 in. After steam sterilizing at 121°C for 30 minutes at 21 psi and a 72-hr ambient drying period, the cell pack was reinserted in the case and sealed. The cell was filled with 40% KOH and allowed to stand for 84 hr before initiation of electrical performance testing. In addition to the formation charge and letdown discharge, operational charge/discharge cycles were conducted to yield data comparable with the other test cells (see section entitled ELECTRICAL PERFORMANCE).

Test results. - Figures 1 and 2 show average charge/discharge curves for this cell with individual cycle results tabulated. These electrical data are slightly better than those obtained with the 135°C dry heat sterilized cells. From this exploratory data, autoclave sterilization at the higher assembly level shows greater electrical degradation than assembly with the 125°C cellophane.

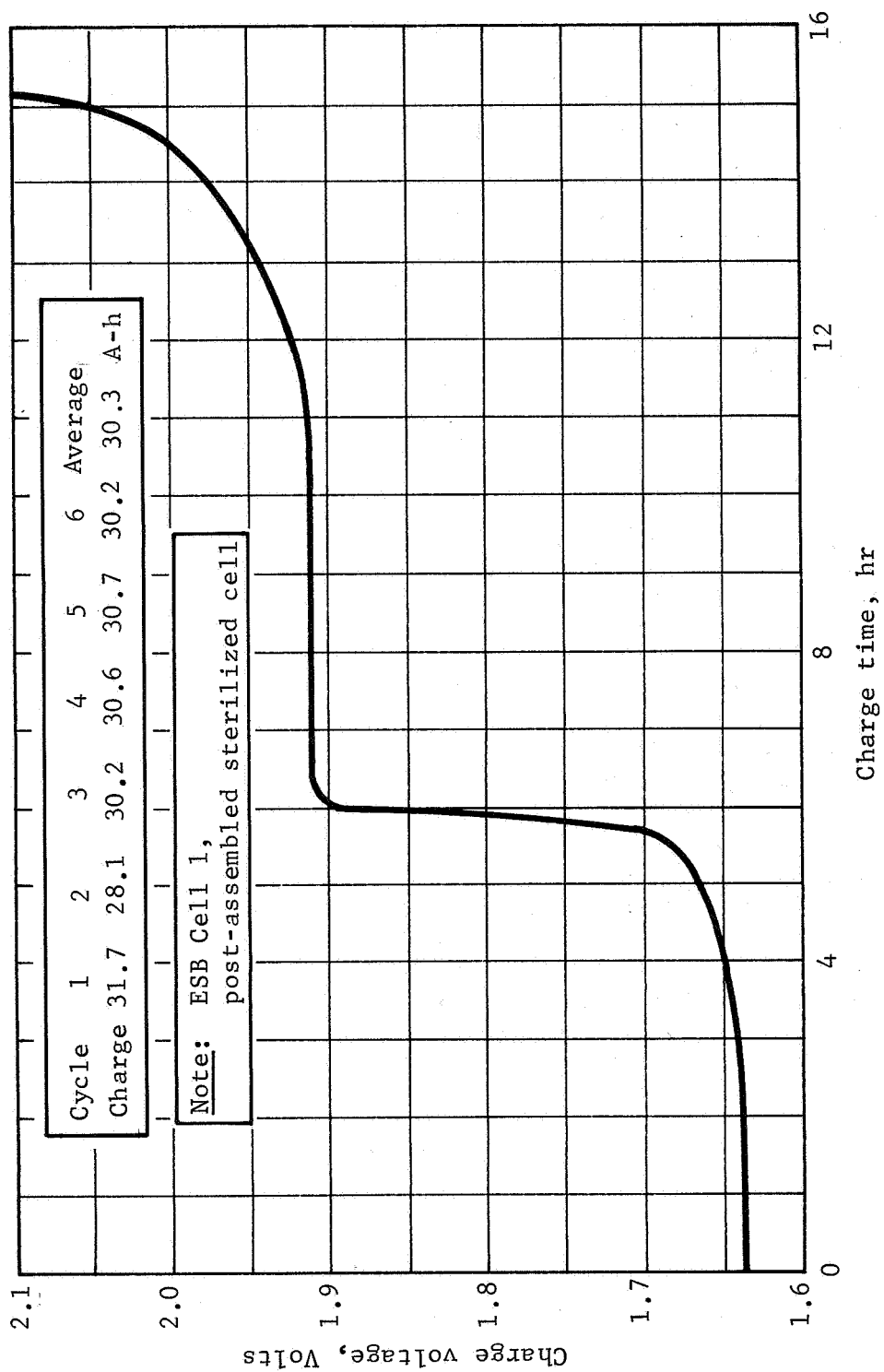


Figure 1.- Average Charge Curve, ESB S-25, 2-A Charge Rate

Cycle	1	2	3	4	5	6	Average	
Discharge	27.2	26.9	29.1	28.1	25.7	28.0	27.5	A-h

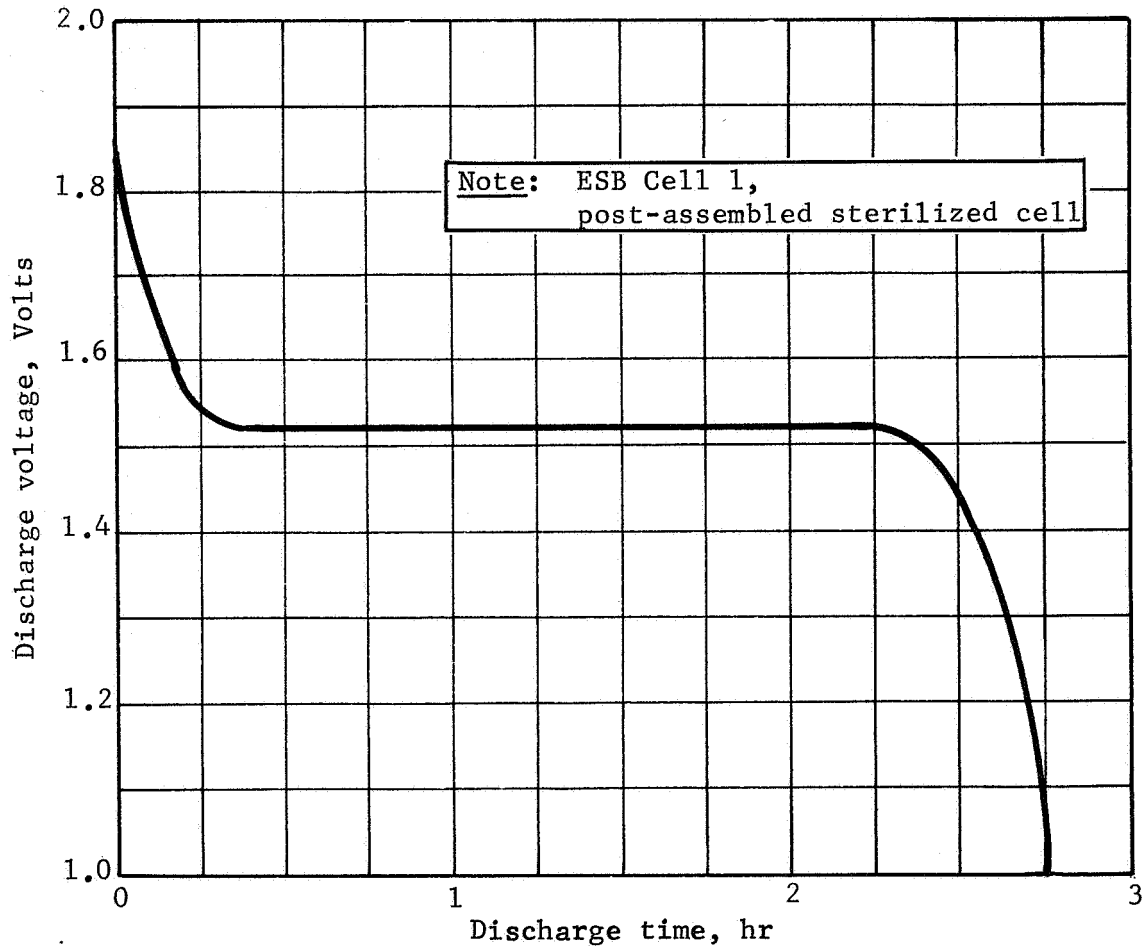


Figure 2.- Average Discharge Curve, ESB S-25, 8-A Discharge Rate

### Conclusions

- 1) All cell components except the nylon case and cover and the cellophane separators can be sterilized at 135°C for 60 hr with no evident degradation;
- 2) Potassium hydroxide and ethyl alcohol can be successfully sterilized with dry heat at 135°C when sealed in a stainless steel cylinder;
- 3) The nylon case and lid show dimensional shrinkage after dry heat sterilization but recover to meet the original tolerances when subjected to high humidity conditions;
- 4) Cellophane separators exhibit darkening, embrittlement, and shrinkage after dry heat sterilization but analysis of test data shows no statistically significant changes in tensile properties after autoclaving or dry heat followed by long ambient exposures;
- 5) All materials and components are amenable to autoclave sterilization procedures, but dimensional shrinkage occurs with the cellophane separator material;
- 6) Suitable sterilization procedures are available to permit component passclave sterilization as part of a sterile assembly process for batteries.

## ASSEMBLY OPERATIONS

The merits of a sterile assembly operation must be evaluated on the basis of its impact on standard assembly procedures. Particular attention must be given to the effects on the efficiency and attitudes of assembly personnel and the modifications from standard tooling and fixtures. Recognizing these implications, a planned program was initiated that involved a sequence of three types of assemblies. Initial assembly (standard cells) used parts and operating procedures based on the battery manufacturer's standard procedures. Modifications were made to these standard procedures that recognized the forthcoming use of this method under sterile isolator conditions. This phase concurrently permitted evaluation of the procedure and assurance of adequate operator training. In turn, the second phase (control cells) used the same assembly operations but substituted cell components that had been subjected to a sterilization cycle. The third phase (sterile cells) imposed a total sterile operation using the same assembly procedure with sterile components in a sterile isolator environment (see fig. 3).

The following subsections describe standard cell assembly operation, control cell assembly, and the sterile cell assembly. Conclusions are given at the end of the section.





## Standard Cells

The procedure used in the assembly of cells was developed from the "Assembly Procedure for ESB/EMED 7-11000-5 Dry Uncharged Cell," provided by ESB Inc. The step by step procedure will be found in Appendix C of this report. Before the actual assembly of the standard cells, the assembly process and tooling were verified by the assembly technicians using sample cell components. Concurrently, this verification served to familiarize the technicians with the total assembly sequence.

Assembly of the 12 standard cells was performed on an open bench. The cells selected for this program were the ESB/EMED 7-11000-5 S-25 silver-zinc secondary cell. This cell has a minimum capacity rating of 25 A-h when discharged at 1-hr rate. This type cell is regularly manufactured by the Exide Missile and Electronics Division of ESB, Inc, who supplied the cell components for this program. The cell components are described in Appendix C of this report.

During the review and verification of the standard assembly procedure provided by ESB, Inc, several areas of improvement were identified and are described below.

Cell assembly drawing error. - During the assembly of the sample cells it was noted that these were an error in the ESB assembly drawing, which would have made it impossible to seat the cell cover after the cell pack was in place. ESB was contacted to check their engineering. The corrected dimensions as well as a revised engineering drawing were supplied by ESB. The procedure and tooling was changed and verified before proceeding.

Improved soldering techniques. - In reviewing the restrictions imposed by operation in the closed environment of an isolator system, it was apparent that the use of a 200-W soldering iron recommended by ESB for terminal soldering operations would present a possible hazard. Investigations indicated that a low-voltage, carbon-resistance, soldering tool would provide satisfactory terminal soldering. An appropriate tool was constructed, which could withstand the sterilization environment, and was used for all soldering operations on the program.

Nylon cell case sealing. - The cell case/cover sealing process used on the cell assembly line by ESB was not suitable for isolator use. Therefore, it was necessary to develop an isolator adaptable sealing technique for the nylon cell cases.

Table 5 lists the experimental sealants investigated and the results obtained using 1x $\frac{1}{2}$ -in. nylon test strips. Adhesions were obtained with sealant formulations C and D. To verify their adequacy for cell assembly usage, two nylon cell cases/lids were bonded with each formulation, cured at 25°C for 12 hr, and tested at 15 psi internal pressure. No failures of the bonds occurred under these conditions. A decision was made to use sealant C in the assembly operations because of the more caustic nature of sealant D.

TABLE 5.- NYLON CELL CASE SEALANTS

Sealant	Compound	Set time/ temperature	Results
A	2 g 828 epoxy 2 g V-40 .25 g 80% ethanol .25 g Resorcinol	12 hr/25°C	No bond
B	2 g 828 epoxy 2 g V-40 .05 g 80% ethanol .05 g Resorcinol	12 hr/25°C	No bond
C	5 g 80% ethanol 5 g Resorcinol	12 hr/25°C	Bonded
D	5 g 88% phenol	12/hr/25°C	Bonded

No other difficulties were encountered in the assembly of the standard cells.

#### Control Cells

Cell components for the 12 control cells were prepared for sterilization and subjected to a sterilization cycle as described in Appendix C.

The assembly of these cells was accomplished on an open bench with the same procedure used in the assembly of the standard cells. No difficulties were encountered in assembly of these cells.

## Sterile Assembly

Sterile assembly of the cells required that (1) the equipment and materials be sterilized before assembly; (2) the entire assembly process be conducted in a presterilized atmosphere isolated from the operator; (3) rigid procedural controls be instituted to avoid introducing contamination, and (4) biological monitoring be conducted to detect breaches of security. The total procedures for accomplishing these are in Appendix C and E of this report.

Assembly of the sterile assembled cells was accomplished within the controlled environment of the sterile isolator system by the same technicians who assembled the cells in the open bench phases of the program. The cells were assembled using the same procedure used for assembly of the standard (nonsterile) cells and the control (sterile/nonsterile) cells.

The first 12 of the sterile assembled cells were constructed using cellophane separator material that had been dry heat sterilized at 135°C for 60 hr. The cellophane showed considerable darkening in color and was difficult to handle because of brittleness of the cellophane. The humidity of the isolator system was raised by admitting steam from the passclave into the isolator system. With the humidity at 35 to 40%, the cellophane regained a flexibility permitting folding without cracking of the film. The remaining 12 cells were assembled with cellophane sterilized at 125°C for 60 hr to provide comparative data with the 135°C units.

## Conclusions

Based on a review of the data obtained from this portion of the program, the following conclusions have been reached:

- 1) No apparent difficulties were noted in the assembly of silver-zinc cells of the S-25 type in a sterile isolator environment;
- 2) The assembly of sterile silver-zinc cells or batteries, in properly designed isolator systems, is feasible;
- 3) Use of a high relative humidity in the isolator system rectified the handling problems with the dry heat sterilized cellophane. Because high relative humidity can introduce adverse effects on some materials, further investigation is warranted to eliminate this constraint on the use of cellophane separators;
- 4) The efficiency of the technicians assigned to this program was not hampered materially by the constraints of the isolator system.

## ELECTRICAL PERFORMANCE

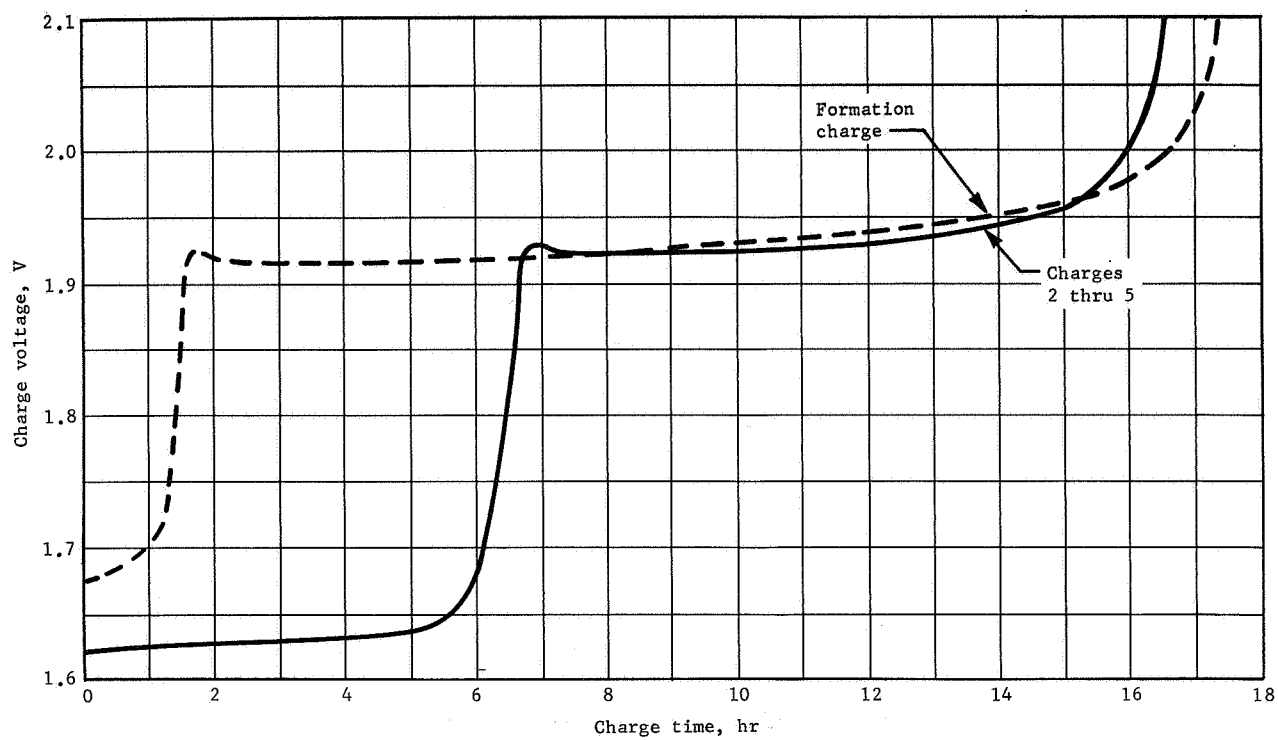
The feasibility of sterile assembly of batteries depends on the impact of this mode of assembly on the performance characteristics of the batteries. Of primary interest is any evidence of degradation in electrical performance. Accordingly, a sequence of electrical performance tests was conducted to obtain data permitting comparative analysis of the standard, control, and sterile assembled cells. This section describes the electrical testing and the data obtained; compares the performance of the standard, control, and sterile cell groups; and presents conclusions based on this electrical performance data.

### Technical Approach

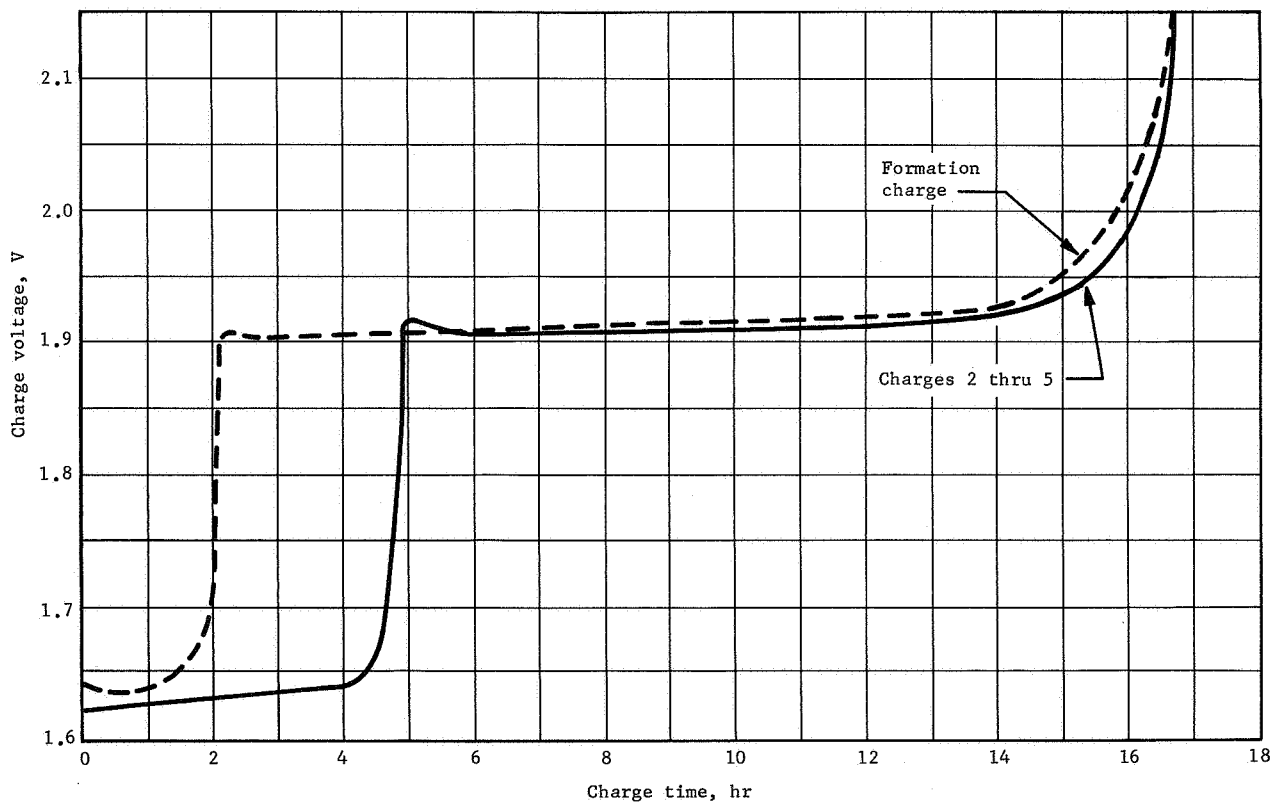
Following assembly and filling with electrolyte, each cell was electrically cycled on an individual basis in accordance with the general instructions provided by ESB. Each cell received a formation charge and a letdown discharge within 3 to 14 days after activation followed by four additional charge/discharge cycles.

Cell charging. - Each cell was formation charged at 2.0 A constant current to an end of charge voltage of 2.1 V. The subsequent operational charge procedure was identical to the formation charge. Figure 4 represents typical charge voltage vs time curves for the standard, control, and sterile cell groups. The significantly different characteristics of the cell during formation charge and the subsequent operational charges is indicated by the broken and solid lines. The formation charge is required to stabilize the operation of a new cell and the differences are normal characteristics for cell performance. The cell charging procedure and the charging circuit are described in Appendix D.

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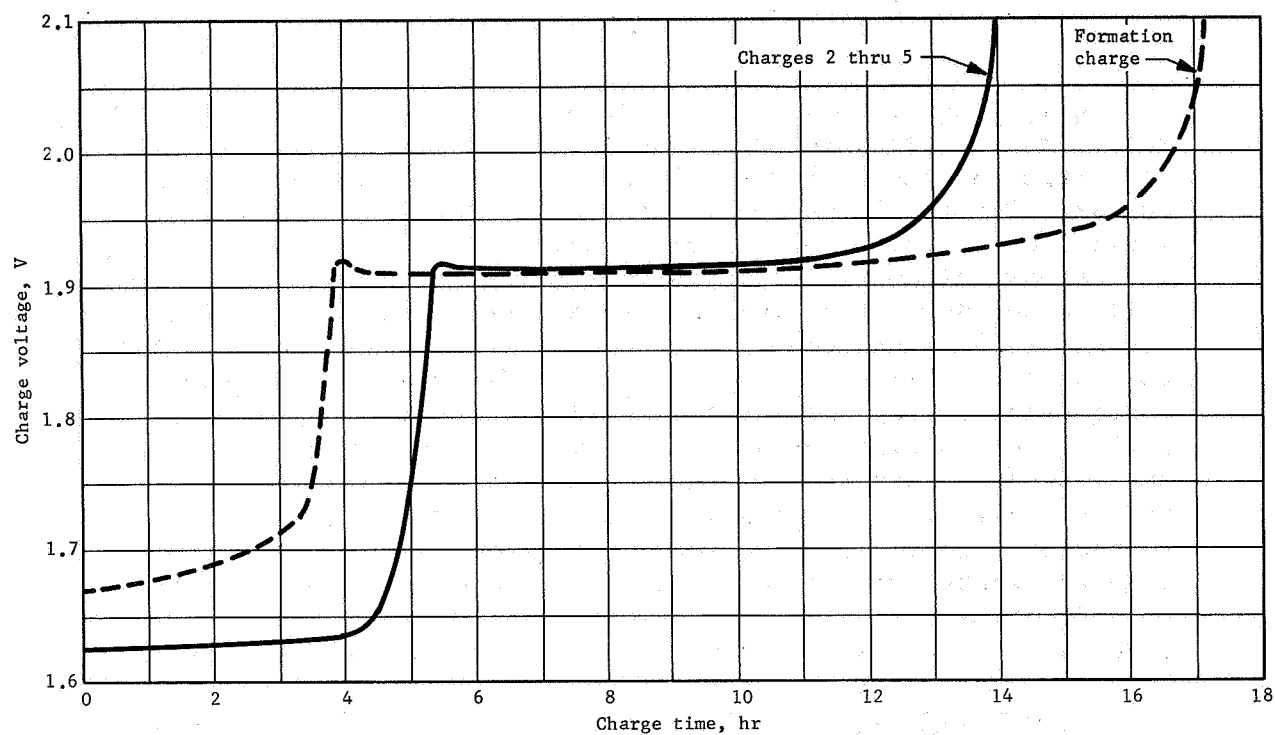


(a) Cells 1 thru 12 (standard cells)

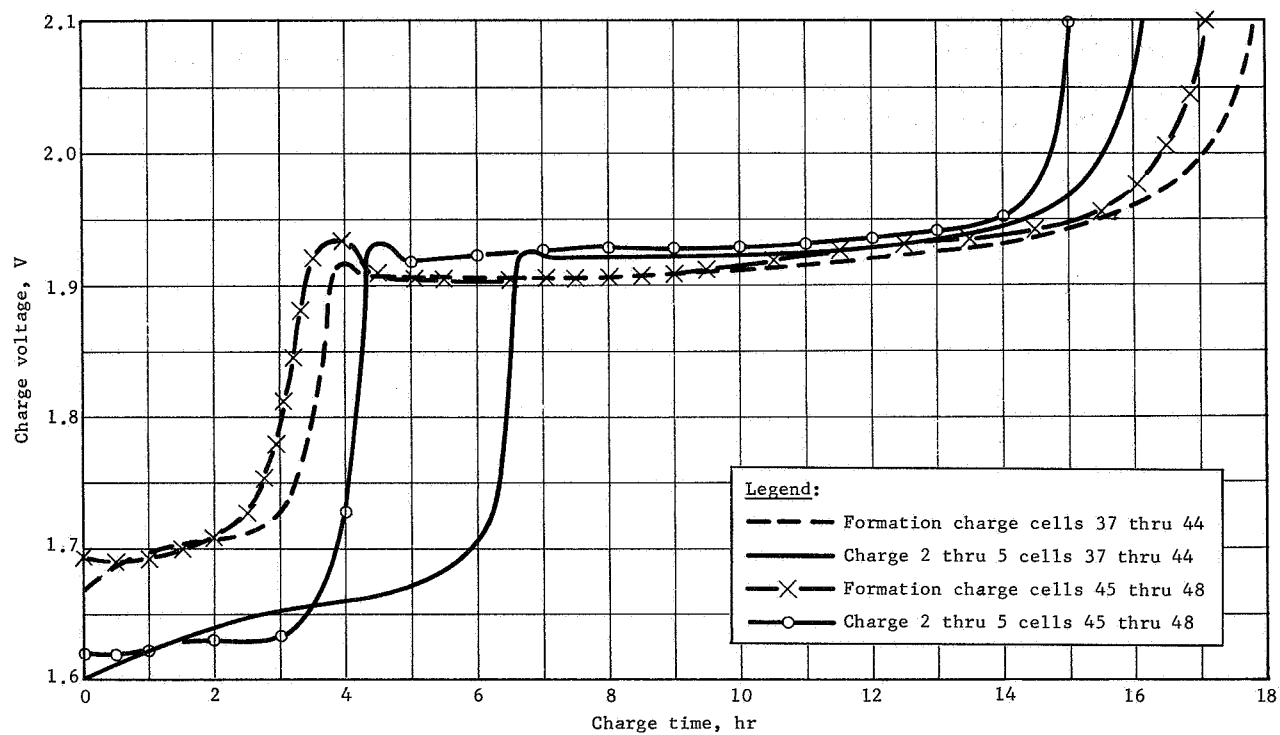


(b) Cells 13 thru 24 (control cells)

Figure 4.- Average Charge Curves, 2-A Rate



(c) Cells 25 thru 36 (sterile cells with 135°C cellophane)



(d) Cells 37 thru 48 (sterile cells with 125°C cellophane)

Figure 4.- Concluded



Cell discharging. - The letdown discharge following the formation charge was conducted at 4 A constant current to an end of discharge voltage of 1.0 V. Figure 5 shows the spread in performance for the formation or letdown discharge of the standard, control and sterile cell groups. Similar to the formation charge, the letdown discharge variations are normal characteristics for cell performance. The initial operational discharges were established at 25 A constant current (the 1 hr rate for this cell) to an end of discharge voltage of 1.0 V. Experience with this discharge rate during the first operational discharge (cycle 2) of cells 1 thru 4 ;indicated that the rapid change in voltage near the end of discharge made the determination of the discharge time to 1.0 V difficult to measure accurately. This voltage change characteristic is clearly shown in figure 6(a). The discharge rate was therefore changed to 10 A to reduce the rate of change of cell voltage near the end of discharge. The cell discharging procedure and the discharging circuit are described in Appendix C.

Data recording. - During each cell electrical test cycle the charge and discharge current, the cell terminal voltage, and time were recorded at regular intervals of not more than 1 hr and near the end of charge or discharge the recordings were taken at 5 to 10 minute intervals. The data were recorded manually by the technicians conducting the tests from readings on both analog and digital instrumentation.

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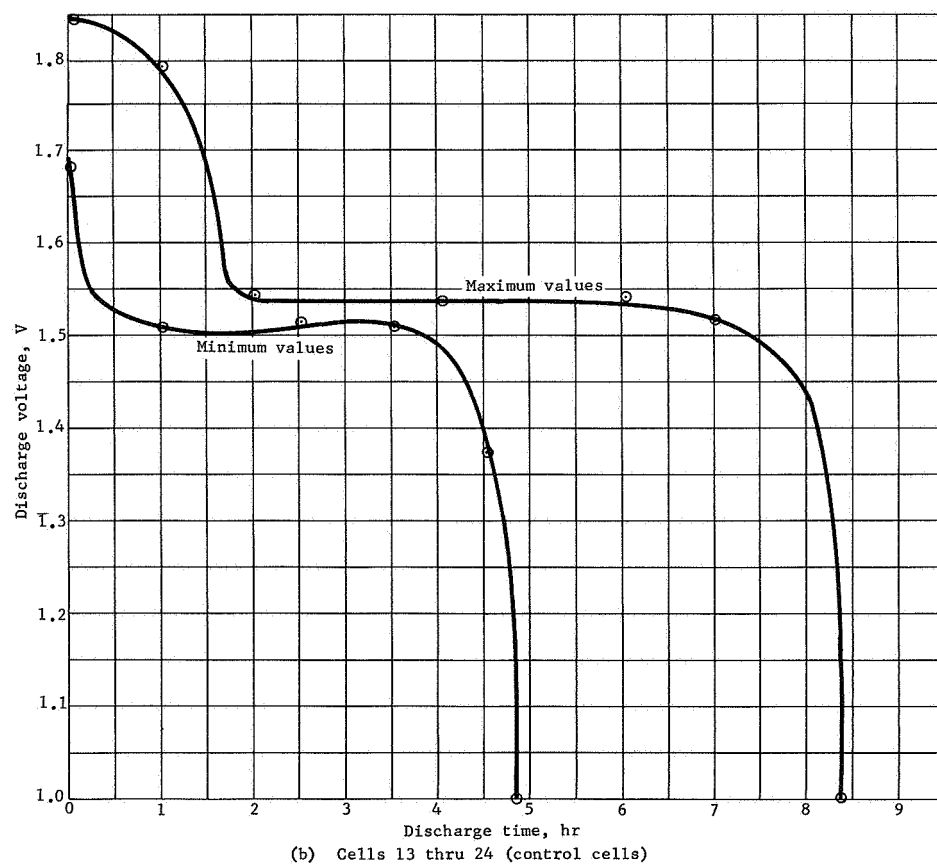
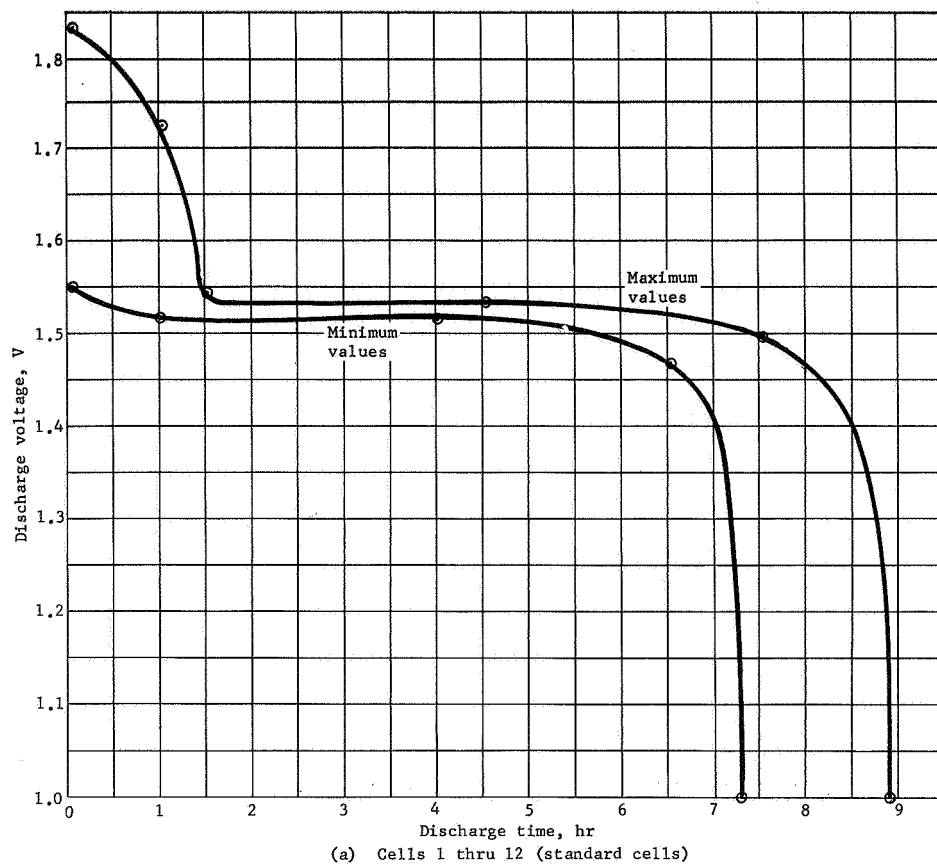
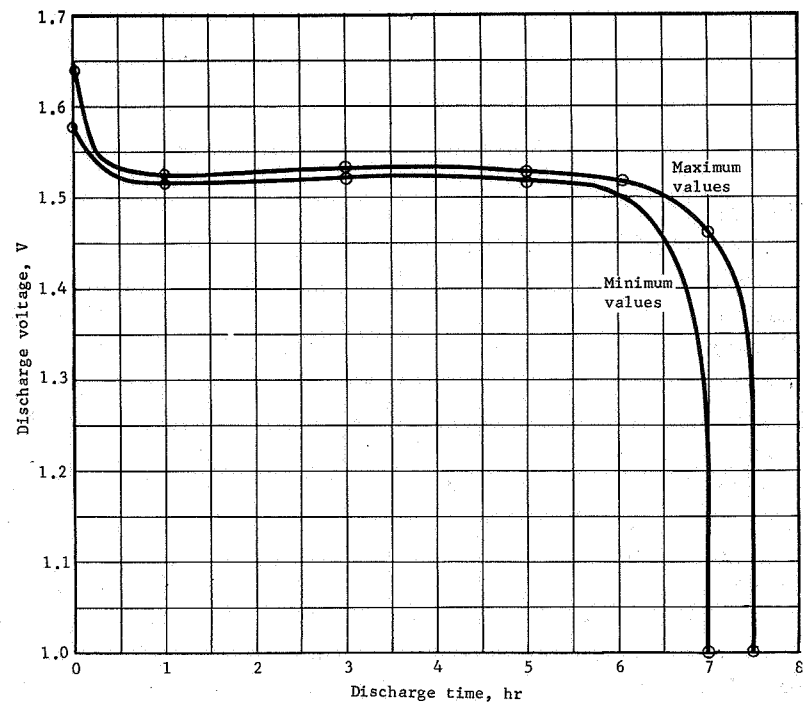
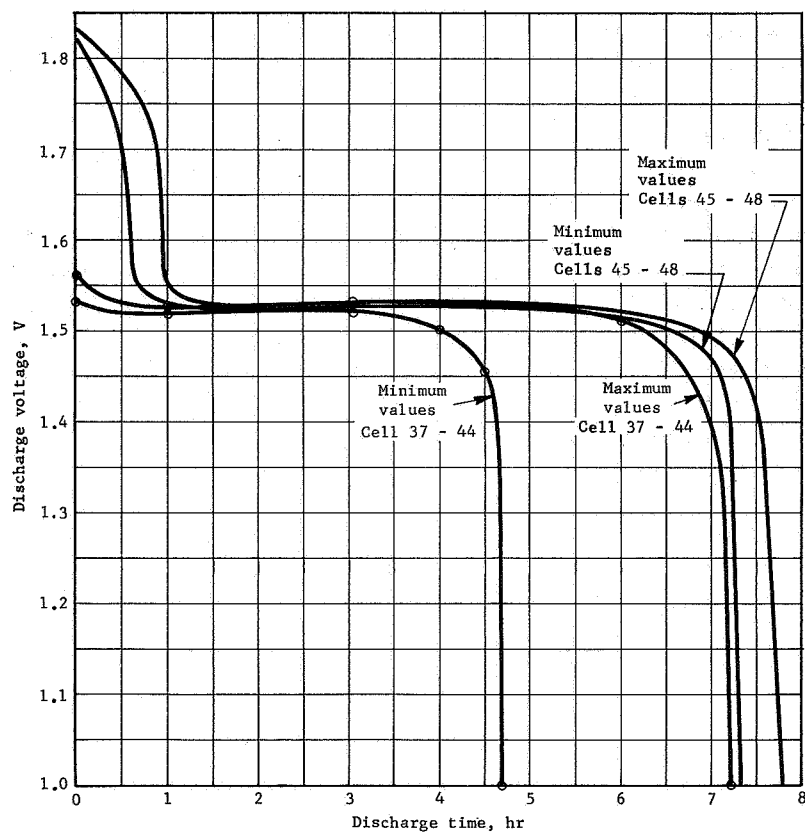


Figure 5.- Letdown Discharge Performance Curves, 4-A Rate



(c) Cells 25 thru 36 (sterile cells with 135°C cellophane)



(d) Cells 37 thru 48 (sterile cells with 125°C cellophane)

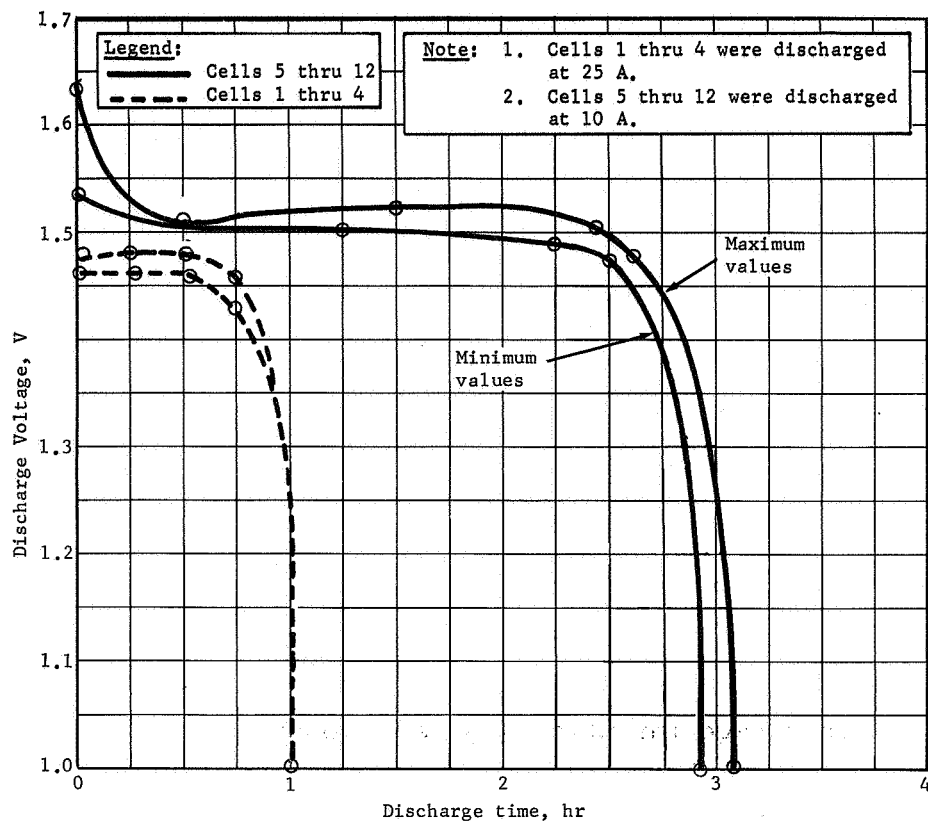
Figure 5.- Concluded

### Standard Cell Performance

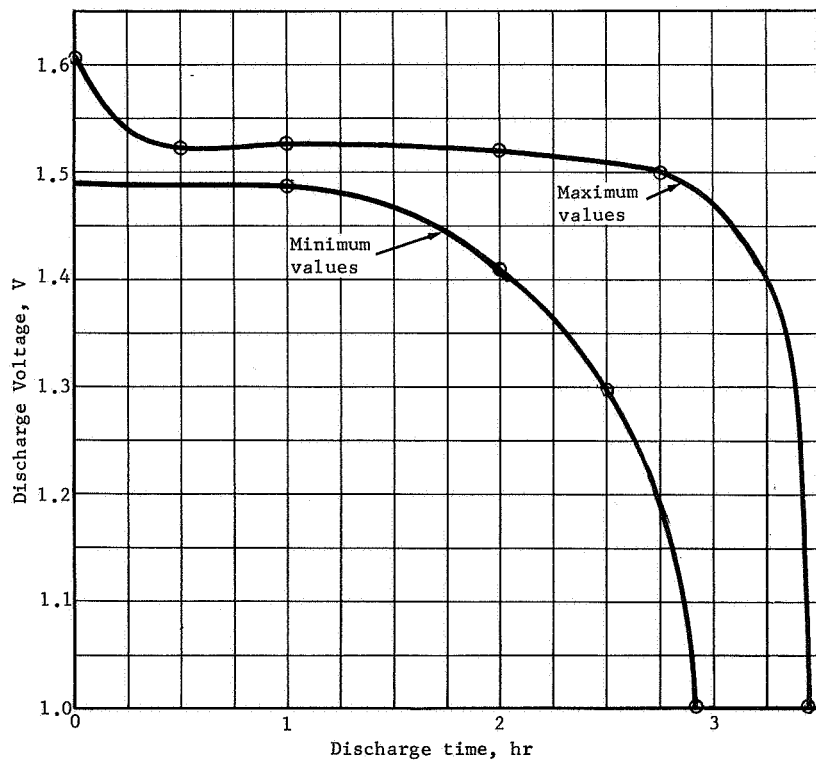
The cell discharge voltage vs time data for the standard cells (1 thru 12) is shown for cycles 2, 3, 4, and 5 in figure 6. The two solid line curves in this figure indicate the spread in performance during cycles 2, 3, 4, and 5. The discharge data for cycle 2 shows the 25 A discharge of cells 1 thru 4 separately from the 10 A discharge of cells 5 thru 12. The cell voltage during the fifth discharge cycle varied from 1.52 to 1.48 V or a difference of approximately 40 mV during the early portion of the discharge at the  $\text{Ag}_2\text{O}$  level of the positive electrode before the rapid voltage drop associated with the end of discharge. The end of discharge time to 1.0 V varied from 3.55 to 3.17 hr or a difference of 0.38 hr. At the 10-A discharge rate, this represents a 3.8 A-h spread during a total of 12 discharges. The broken line in figure 6(d) presents the average discharge curve for the 12 standard cells during the fifth cycle, indicating that the average  $\text{Ag}_2\text{O}$  discharge voltage was approximately 1.51 V and the discharge capacity was 33.0 A-h.

The data in table 6 summarize the charge and discharge ampere-hours for the standard cell group. Average values for the ampere-hours accepted on charge and delivered on discharge are shown for each cell for cycles 2 thru 5 and for all cells for each cycle. Cycle efficiency numbers based on the charge and discharge ampere-hours are also included. The performance numbers for the standard cell group are summarized in the lower right-hand portion of the table and indicate that the average values for charge and discharge are 32.8 and 31.4 A-h, respectively, with an efficiency of 95.7%. The discharge data obtained during the letdown discharge (cycle 1) at 4 A for all 12 cells and the cycle 2 discharge at 25 A for cells 1 thru 4 have been excluded from these averages.

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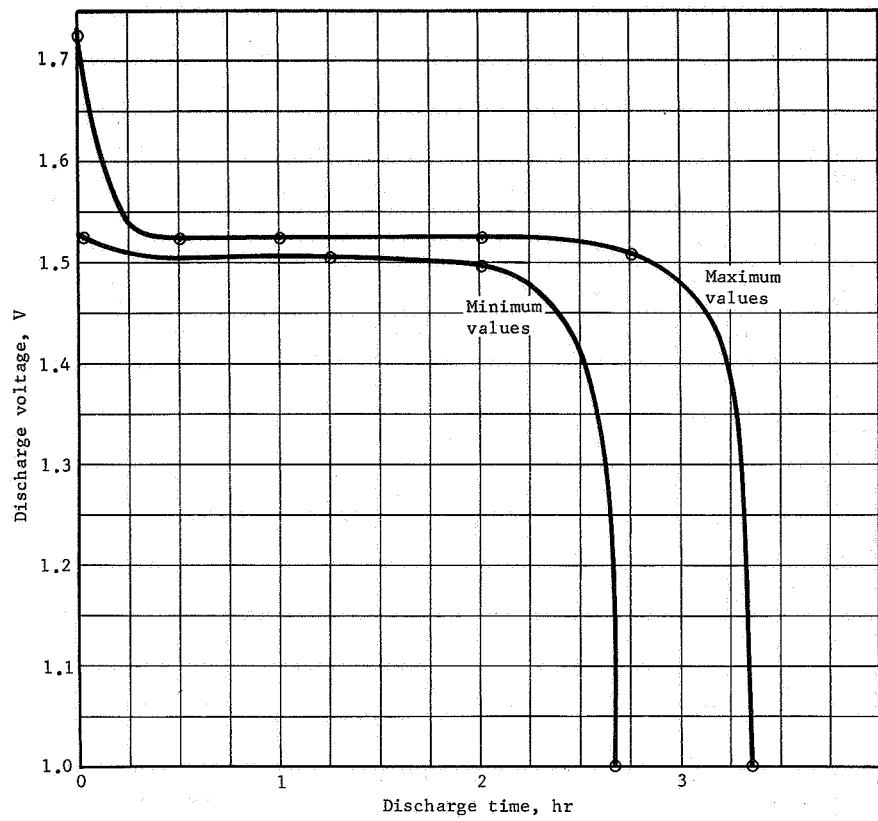


(a) Cycle 2 discharge, composite of cells 1 thru 4 and 5 thru 12

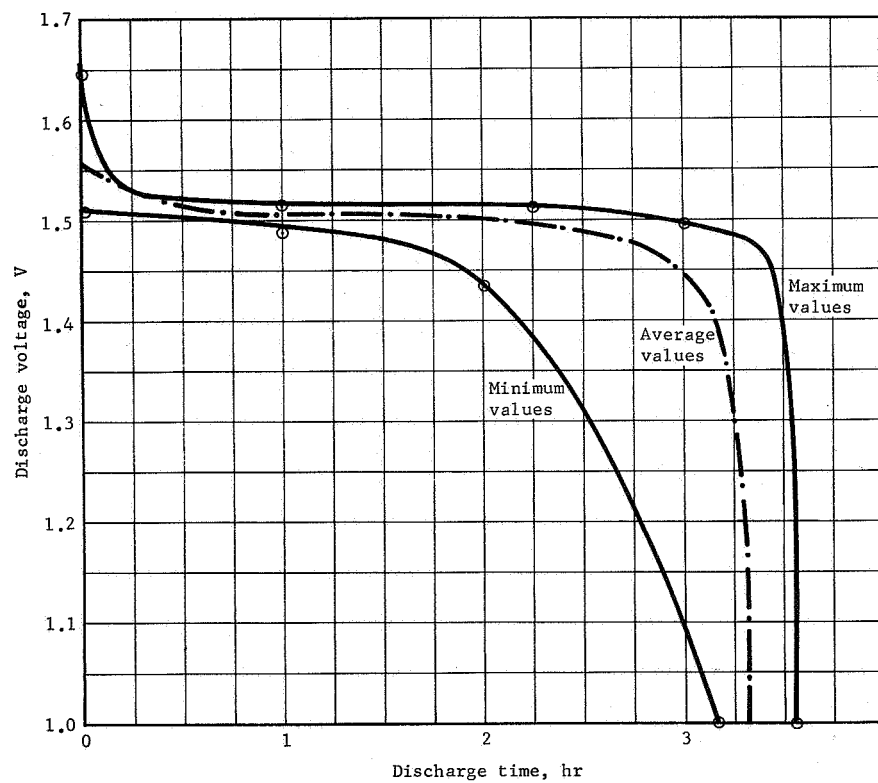


(b) Cycle 3 discharge, cells 1 thru 12

Figure 6.- Standard Cell Discharge Curves, 10-A Rate



(c) Cycle 4 discharge, cells 1 thru 12



(d) Cycle 5 discharge, cells 1 thru 12

Figure 6.- Concluded



TABLE 6.- STANDARD CELL GROUP CHARGE AND DISCHARGE AMPERE-HOURS

Cell	Cycle 1 <sup>a</sup>			Cycle 2			Cycle 3			Cycle 4			Cycle 5			Cell average		
	Charge	Dis-charge	Efficiency, %	Charge	Dis-charge	Efficiency, %	Charge	Dis-charge	Efficiency, %	Charge	Dis-charge	Efficiency, %	Charge	Dis-charge	Efficiency, %	Charge	Dis-charge	Efficiency, %
1	35.0	32.7	93.4	28.8	<sup>a</sup> 25.5	88.5	34.8	33.7	96.8	30.5	28.7	94.1	33.5	32.5	97.0	32.9	31.6	96.0
2	34.1	33.7	98.8	28.8	<sup>a</sup> 25.5	88.5	34.8	33.3	95.7	30.5	28.7	94.1	34.1	33.7	98.8	33.1	31.9	96.4
3	36.9	35.4	95.9	28.0	<sup>a</sup> 25.5	91.1	35.5	34.2	96.3	31.4	29.7	94.6	34.6	34.3	99.1	33.8	32.7	96.7
4	36.1	33.7	93.4	28.2	<sup>a</sup> 25.5	90.4	33.3	31.7	95.2	28.5	26.7	93.7	32.8	31.7	96.7	31.5	30.0	95.2
5	31.5	31.1	98.7	30.5	29.7	97.4	32.6	32.2	98.8	34.1	32.3	94.7	34.6	33.8	97.7	33.0	32.0	97.0
6	32.8	32.5	99.1	31.4	29.7	94.6	35.4	33.0	93.2	34.8	33.5	96.3	34.8	35.5	<sup>a</sup> 102	33.9	32.1	94.7
7	32.8	32.5	99.1	30.9	29.7	96.1	32.6	32.2	98.8	33.8	32.5	96.2	34.3	33.3	97.1	32.9	31.9	97.0
8	35.1	34.9	99.4	31.8	30.5	95.9	33.5	32.0	95.5	34.1	32.8	96.2	35.3	35.5	<sup>a</sup> 101	33.1	31.8	96.1
9	35.7	33.0	92.4	32.7	30.2	92.4	32.0	30.8	96.3	33.0	33.3	<sup>a</sup> 101	32.8	32.5	99.1	31.7	31.2	98.4
10	34.8	29.1	83.6	32.7	30.2	92.4	32.0	30.8	96.3	33.0	33.3	<sup>a</sup> 101	33.7	32.5	96.4	32.8	31.2	95.1
11	33.8	31.7	93.8	31.3	28.8	92.0	30.7	29.2	95.1	31.8	32.0	<sup>a</sup> 101	33.7	32.5	96.4	31.9	30.2	94.7
12	35.2	32.5	92.3	32.7	30.2	92.4	32.0	30.5	95.3	32.5	33.3	<sup>a</sup> 102	32.5	32.5	<sup>a</sup> 100	32.4	30.4	93.8
Cycle average	34.5	32.7	94.8	31.8	29.9	94.1	33.3	32.0	96.1	32.2	30.6	95.0	33.8	33.0	97.6			
Cell group average																32.8	31.4	95.7

<sup>a</sup>Indicates data excluded from average values.

### Control Cell Performance

The cell discharge voltage vs time data for the control cells (13 thru 24) is shown for cycles 2, 3, 4, and 5 in figure 7. The two solid curves in this figure indicate the spread in performance during cycles 2, 3, 4, and 5. The cell voltage during the fifth discharge cycle varied from 1.54 to 1.50 V or a difference of approximately 40 mV during most of the discharge at the  $\text{Ag}_2\text{O}$  level of the positive electrode before the rapid voltage drop at the end of discharge. The end of discharge time to 1.0 V varied from 3.39 to 3.28 hr or a difference of 0.11 hr. At the 10-A discharge rate, this represents a 1.1 A-h spread during a total of 12 discharges. The broken line in figure 7(d) presents the average discharge curve for the 12 control cells during the fifth cycle indicating that the average  $\text{Ag}_2\text{O}$  discharge voltage was approximately 1.52 V, and the discharge capacity was 33.6 A-h.

The data in table 7 summarize the charge and discharge ampere-hours for the control cell group. Average values for the ampere-hours accepted on charge and delivered on discharge are shown in the same manner as previously described for the standard cell group. The performance numbers for the control cell group are summarized at the lower right of the table and indicate that the average values for charge and discharge are 33.1 and 32.5 A-h, respectively, with an efficiency of 98.2%. The discharge data obtained during the letdown discharge (cycle 1) at 4 A have been excluded from the average so that the information would be on the same basis of 2 A charge and 10 A discharge as previously presented for the standard cells.

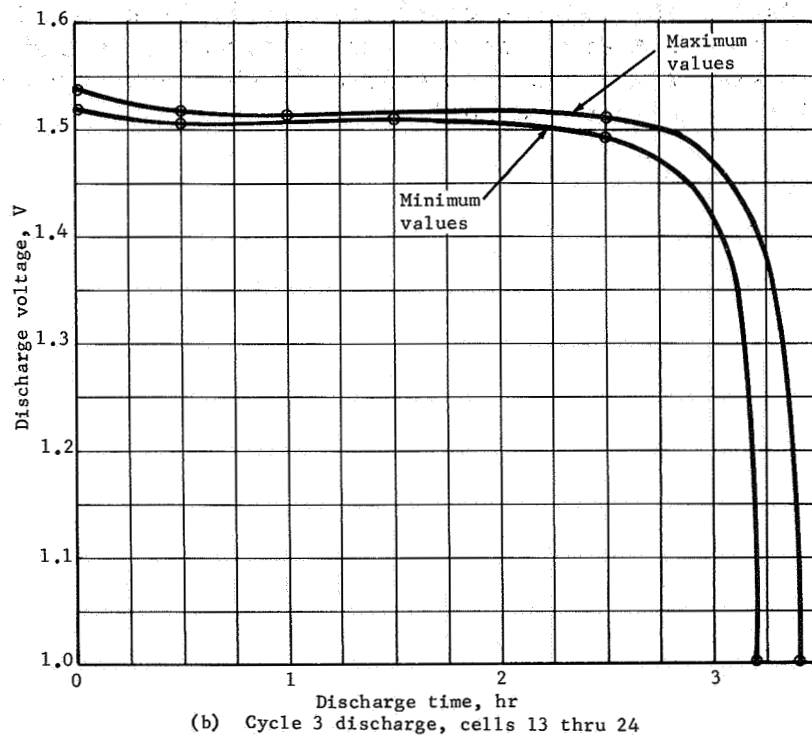
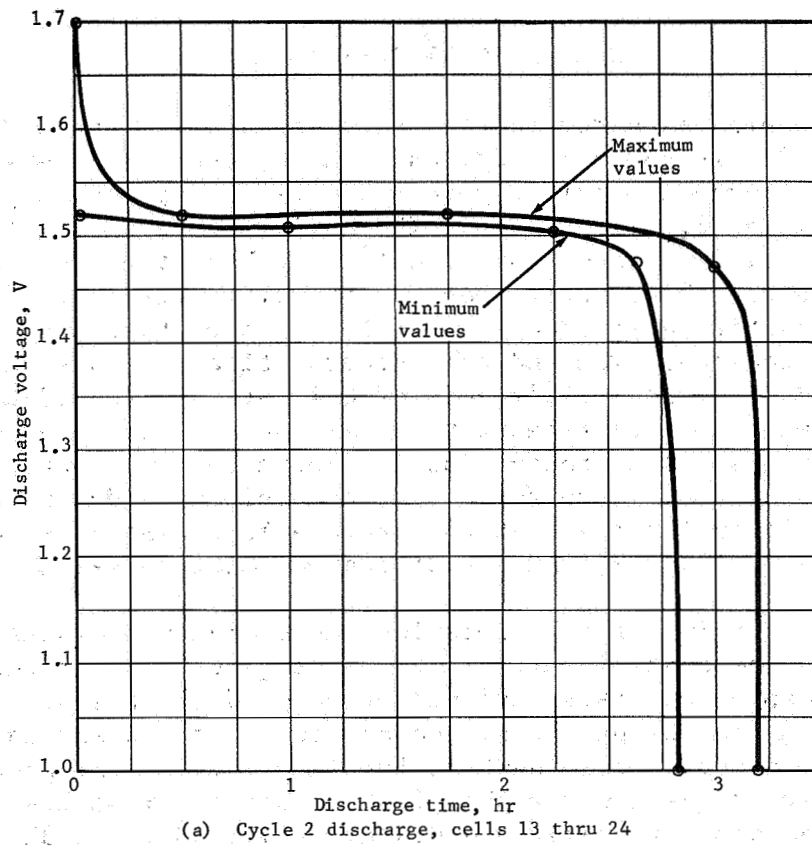
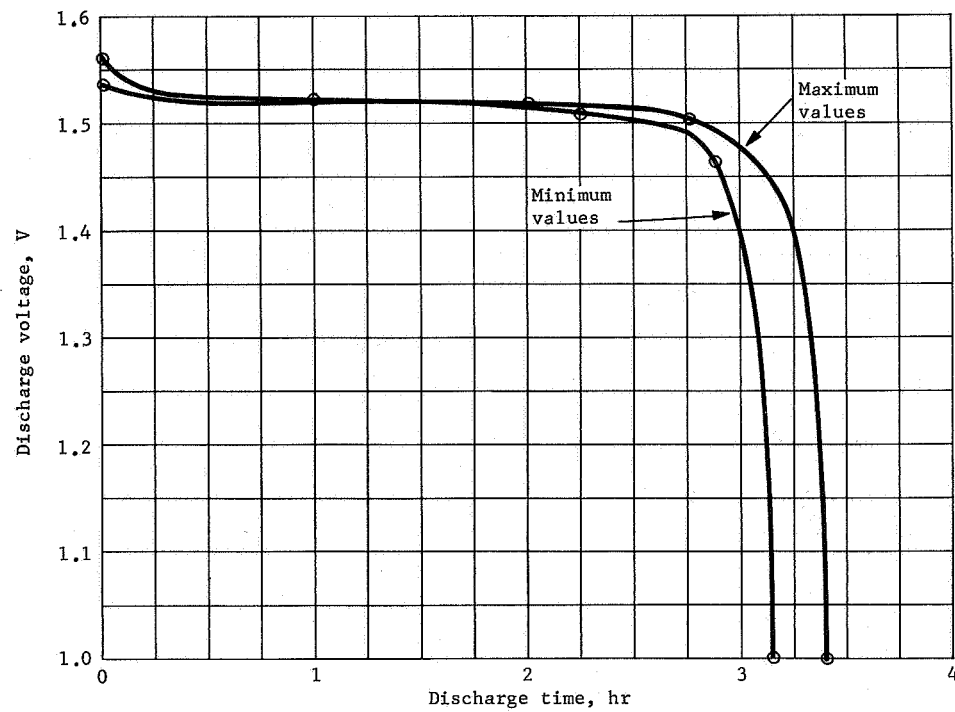
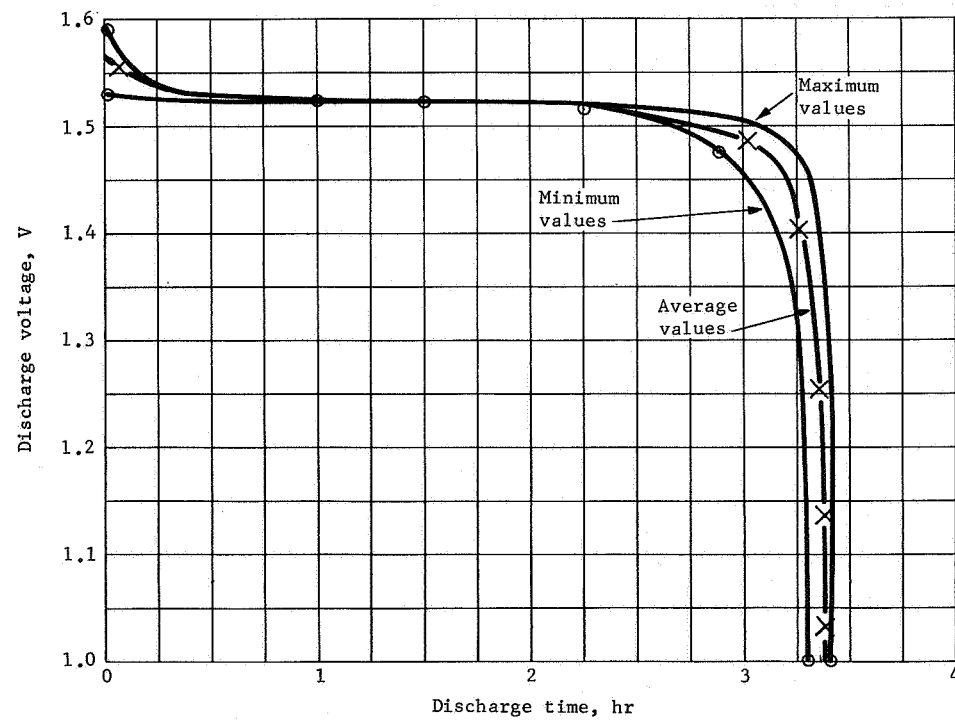


Figure 7.- Control Cell Discharge Curves, 10-A Rate



(c) Cycle 4 discharge, cells 13 thru 24



(d) Cycle 5 discharge, cells 13 thru 24

Figure 7.- Concluded

TABLE 7.- CONTROL CELL GROUP CHARGE AND DISCHARGE AMPERE-HOURS

Cell	Cycle 1 a			Cycle 2			Cycle 3			Cycle 4			Cycle 5			Cell average		
	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %
13	28.3	19.3	68.2	31.5	29.6	94.0	33.4	32.2	96.4	34.7	32.8	94.5	33.4	33.2	99.4	33.3	32.0	96.1
14	31.8	22.0	69.2	29.5	28.3	95.9	32.0	31.7	99.1	33.1	31.3	94.6	32.8	32.8	<sup>a</sup> 100	31.5	30.4	96.5
15	35.0	24.3	69.4	30.7	30.0	97.7	32.9	32.2	97.9	33.8	33.2	98.2	33.4	33.2	99.4	32.7	32.2	98.5
16	32.8	23.0	70.1	30.7	30.0	97.7	33.8	33.3	98.5	34.9	34.0	97.4	33.9	33.6	99.1	33.3	32.7	98.2
17	33.3	23.0	69.1	31.9	30.8	96.6	34.9	33.9	97.1	34.9	34.0	97.4	34.4	33.9	98.5	34.0	33.2	97.6
18	34.8	25.0	71.8	31.9	30.8	96.6	34.3	33.9	98.8	34.9	34.0	97.4	34.2	33.9	99.1	33.8	33.2	98.2
19	33.5	32.6	97.3	32.2	30.6	95.0	32.3	32.2	99.7	33.9	33.2	97.9	33.4	33.2	99.4	33.0	32.3	97.9
20	34.4	33.5	97.4	32.5	30.9	95.1	33.5	33.3	99.4	34.7	34.0	98.0	34.2	33.9	99.1	33.7	33.0	97.9
21	33.2	32.6	98.2	32.5	30.9	95.1	33.4	32.5	97.3	34.7	34.0	98.0	34.2	33.9	99.1	33.7	32.8	97.3
22	31.5	30.4	96.5	33.0	31.9	96.7	33.1	32.5	98.2	34.7	33.8	97.4	33.4	33.2	99.4	33.6	32.9	97.9
23	33.5	32.8	97.9	31.1	29.8	95.8	32.3	32.2	99.7	33.3	32.8	98.5	33.1	33.1	<sup>a</sup> 100	32.2	31.6	98.1
24	34.1	33.5	98.2	30.7	30.0	97.7	32.3	32.2	99.7	33.3	32.7	98.2	33.1	33.1	<sup>a</sup> 100	32.1	31.6	98.4
Cycle aver- age	33.0	27.6	83.6	31.4	30.3	96.1	33.1	32.6	98.5	34.2	33.3	97.3	33.8	33.6	99.2			
Cell group aver- age																33.1	32.5	98.2

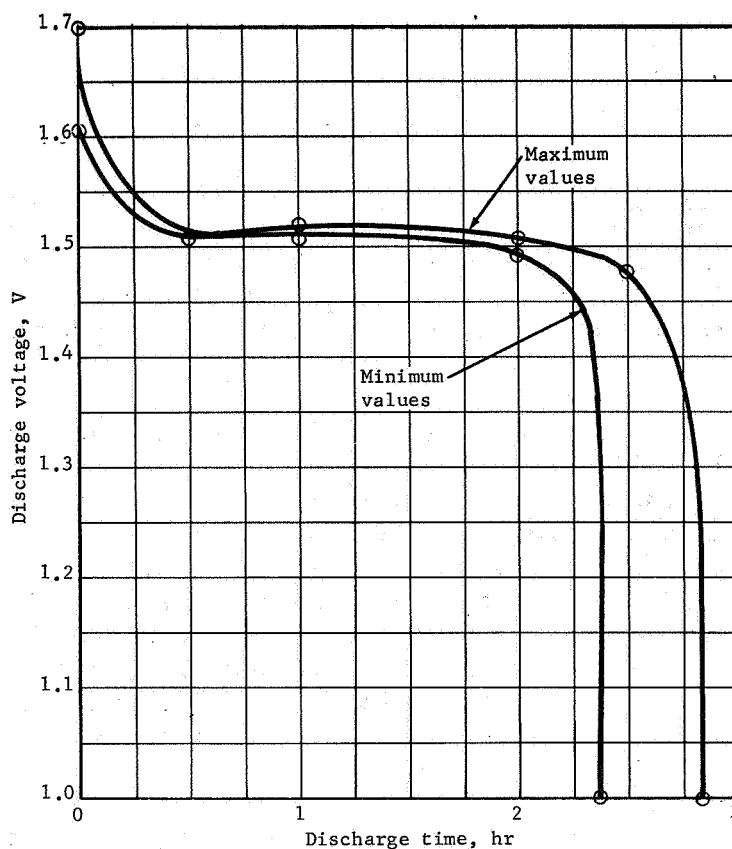
<sup>a</sup>Indicates data excluded from average values.

### Sterile Cell Performance

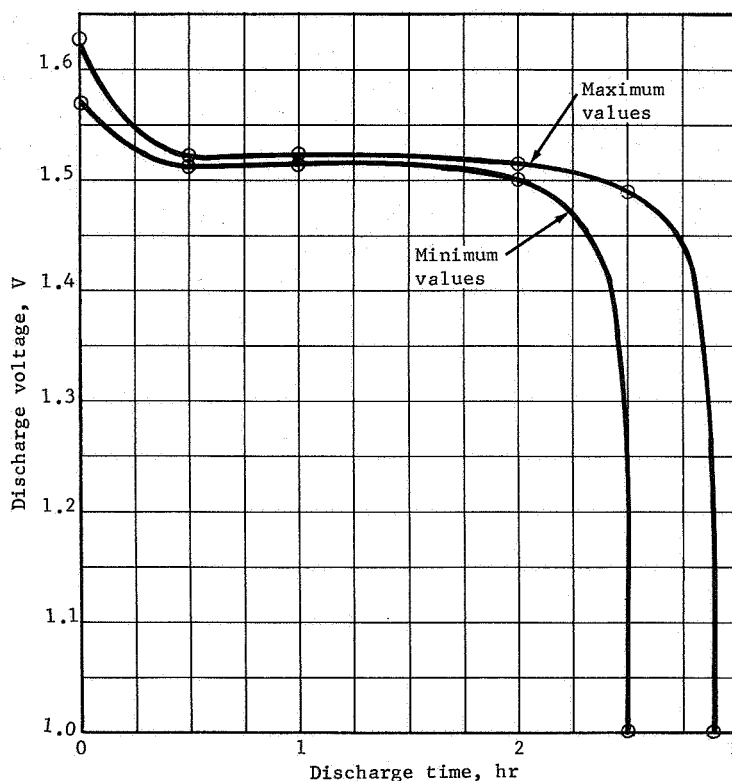
Cells with cellophane sterilized at 135°C. - The cell discharge voltage vs time data for the sterile cells with cellophane sterilized at 135°C (cells 25 thru 36) are shown for cycles 2, 3, 4, and 5 in figure 8. The two solid line curves in this figure indicate the spread in performance during cycles 2, 3, 4, and 5.

The cell voltage during this fifth discharge cycle varied from 1.53 to 1.52 V or a difference of approximately 10 mV during most of the discharge at the Ag<sub>2</sub>O level of the positive electrode before the rapid voltage drop at the end of discharge. The end of discharge time to 1.0 V varied from 2.75 to 1.92 hr or a difference of 0.83 hr. At the 10-A discharge rate, this represents a 8.3 A-h spread during a total of 12 discharges. The broken line in figure 8(d) presents the average discharge curve during the fifth cycle for sterile cells 25 thru 36, indicating that the average Ag<sub>2</sub>O discharge voltage was approximately 1.52 V and the discharge capacity was 25.1 A-h.

The data in table 8 summarize the charge and discharge ampere-hours for the cells with cellophane sterilized at 135°C. Average values for the ampere-hours accepted on charge and delivered on discharge are shown in the same manner as previously described for the standard and control cell groups. The performance numbers for the 135°C group are summarized at the lower right of the table and indicate that the average values for charge and discharge are 28.0 and 26.2 A-h, respectively, with an efficiency of 93.6%. The data obtained during the letdown discharge (cycle 1) at 4 A have been excluded from the average as previously discussed for the standard and control cell groups.

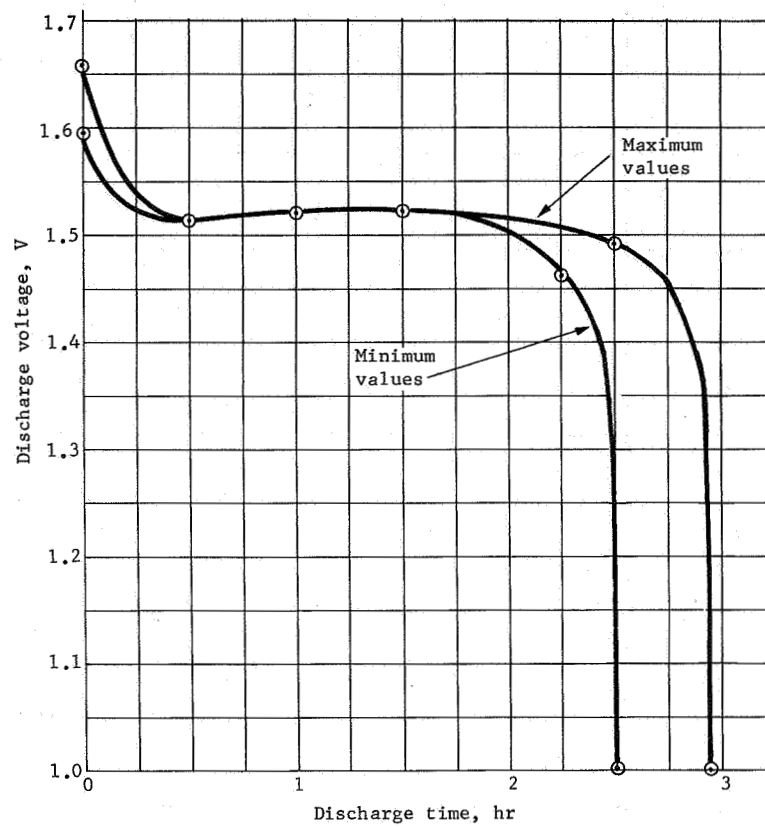


(a) Cycle 2 discharge, cells 25 thru 36

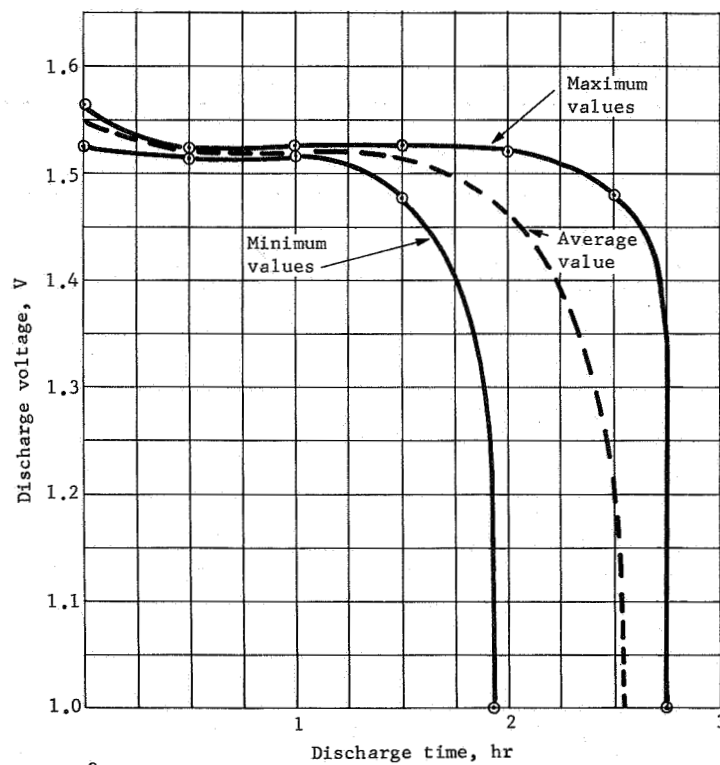


(b) Cycle 3 discharge, cells 25 thru 36

Figure 8.- Sterile Cell Discharge Curves, 10-A Rate (135°C Cellophane Cells)



(c) Cycle 4 discharge, cells 25 thru 36



(d) Cycle 5 discharge, cells 25 thru 36

Figure 8.- Concluded



TABLE 8.- STERILE (135°C CELLOPHANE) CELL GROUP CHARGE AND DISCHARGE AMPERE-HOURS

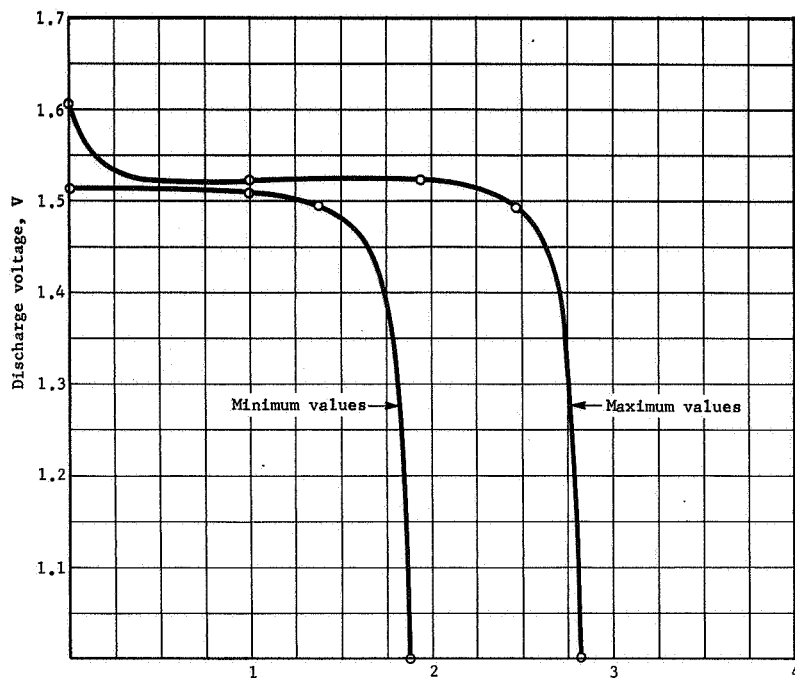
Cell	Cycle 1 <sup>a</sup>			Cycle 2			Cycle 3			Cycle 4			Cycle 5			Cell average		
	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %
25	34.0	28.3	83.2	29.2	28.3	96.9	28.0	26.8	95.7	27.5	27.8	<sup>a</sup> 101	28.4	27.5	96.8	28.3	27.6	96.8
26	33.3	28.3	85.0	28.5	27.5	96.5	27.2	25.7	94.5	26.9	27.0	<sup>a</sup> 100	27.6	25.8	93.5	27.8	26.5	95.3
27	33.2	28.3	85.2	28.8	27.5	95.5	27.5	26.0	94.5	26.9	26.8	99.6	27.6	25.8	93.5	27.7	26.5	95.7
28	33.5	29.0	86.6	28.0	25.0	89.3	27.2	25.3	93.0	26.7	26.4	98.9	26.9	25.0	92.9	27.2	25.4	93.4
29	35.3	29.3	83.0	30.0	26.8	89.3	29.2	28.3	96.9	28.3	28.5	<sup>a</sup> 101	28.1	27.5	97.9	29.1	27.8	95.5
30	34.7	28.0	80.7	28.5	24.5	86.0	27.0	25.8	95.6	26.0	25.7	98.8	25.7	24.2	94.2	26.8	25.1	93.7
31	35.3	28.0	79.3	29.2	24.5	83.9	27.0	25.0	92.6	26.0	25.0	96.2	25.7	24.2	94.2	27.0	24.7	91.5
32	34.7	30.0	86.5	30.0	27.2	90.7	29.7	28.8	97.0	28.8	29.3	<sup>a</sup> 102	28.8	<sup>b</sup>	<sup>c</sup>	29.9	28.4	95.0
33	34.7	29.3	84.4	29.2	25.0	85.6	29.2	25.8	88.4	27.3	26.3	96.3	26.9	19.2	71.4	28.2	24.1	85.5
34	35.3	28.8	81.6	30.0	26.3	87.7	28.8	28.3	98.3	28.2	28.5	<sup>a</sup> 101	27.9	27.5	98.6	28.7	27.7	96.5
35	34.7	28.8	83.0	29.2	24.5	83.9	27.8	26.7	96.0	26.7	26.9	<sup>a</sup> 101	26.7	25.0	93.6	27.6	25.8	93.5
36	34.2	28.3	82.7	29.2	23.7	81.2	27.0	25.8	95.6	26.4	26.3	99.6	26.1	24.2	92.7	27.2	25.0	91.9
Cycle aver- age	34.4	28.7	83.4	29.1	25.9	89.0	28.0	26.5	94.6	26.6	26.1	98.2	27.1	25.1	92.6			
Cell group aver- age													28.0			28.0	26.2	93.6

<sup>a</sup>Indicates data excluded from average values.<sup>b</sup>Indicates cell shorted during open circuit stand.<sup>c</sup>Not calculated.

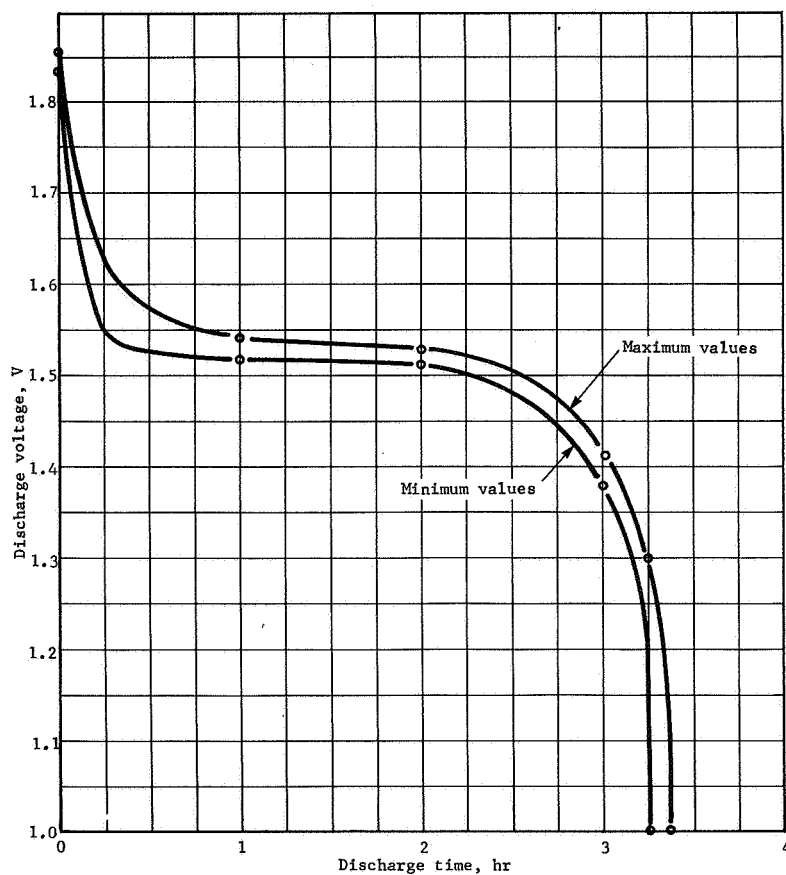
Cells with cellophane sterilized at 125°C. - Immediate addition of electrolyte after assembly of cellophane zinc cells may not always be practiced or desired. Therefore, it appeared to be valuable to ascertain if delayed addition of electrolyte (i.e., storage in a dry condition) would materially affect electrical performance. Cells 37-44 were, therefore activated immediately after assembly; cells 45-48 were stored for 10 weeks prior to electrolyte addition.

125°C sterile cells activated immediately: The cell discharge voltage vs time data for the sterile cells with cellophane sterilized at 125°C and activated immediately (cells 37 thru 44) are shown for cycles 2, 3, 4, and 5 in figure 9. The two solid line curves in this figure indicate the spread in performance during cycles 2, 3, 4, and 5. The cell voltage during the fifth discharge cycle varied from 1.54 to 1.51 V or a difference of approximately 30 mV during most of the discharge at the Ag<sub>2</sub>O level of the positive electrode before the rapid voltage drop at the end of discharge. The end of discharge time to 1.0 V varied from 3.46 to 3.09 hr or a difference of 0.37 hr. At the 10-A discharge rate, this represents a 3.7 A-h spread during a total of 8 discharges. The broken line in figure 9(d) presents the average discharge curve for sterile cells 37 thru 44, indicating that the average Ag<sub>2</sub>O discharge voltage was approximately 1.52 V and the discharge capacity was 32.8 A-h.

The data in upper table 9 summarize the charge and discharge ampere-hours for the cells with cellophane sterilized at 125°C and activated immediately. Average values for the ampere-hours accepted on charge and delivered on discharge are shown in the same manner as previously described for the standard and control cell groups. The performance numbers for the activated 125°C group are summarized at the center right of the table and indicate that the average values for charge and discharge are 32.4 and 31.2 A-h, respectively, with an efficiency of 96.3%. The data obtained during the letdown discharge (cycle 1) at 4 A have been excluded from these averages as previously discussed for the standard and control cell groups.

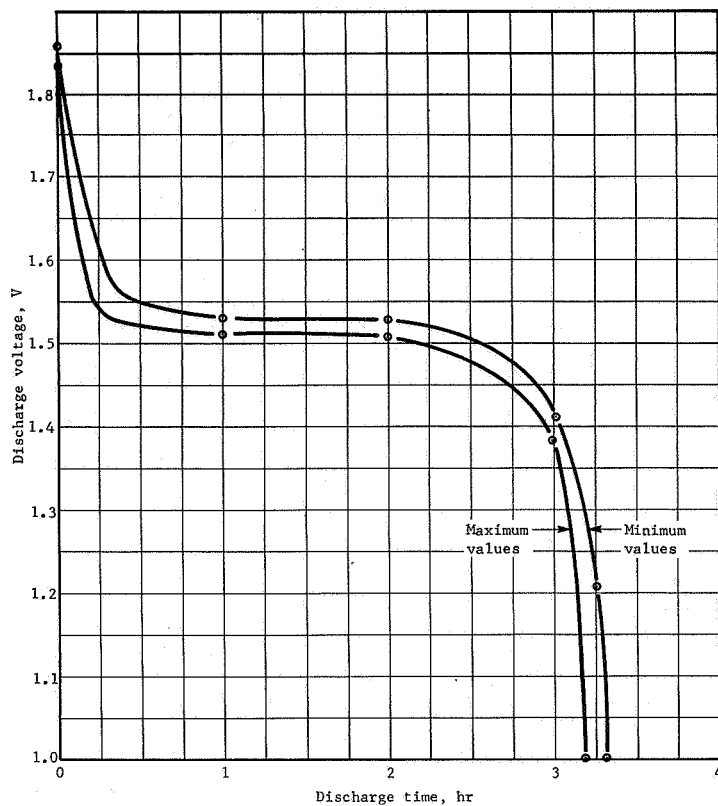


(a) Cycle 2 discharge, cells 37 thru 44

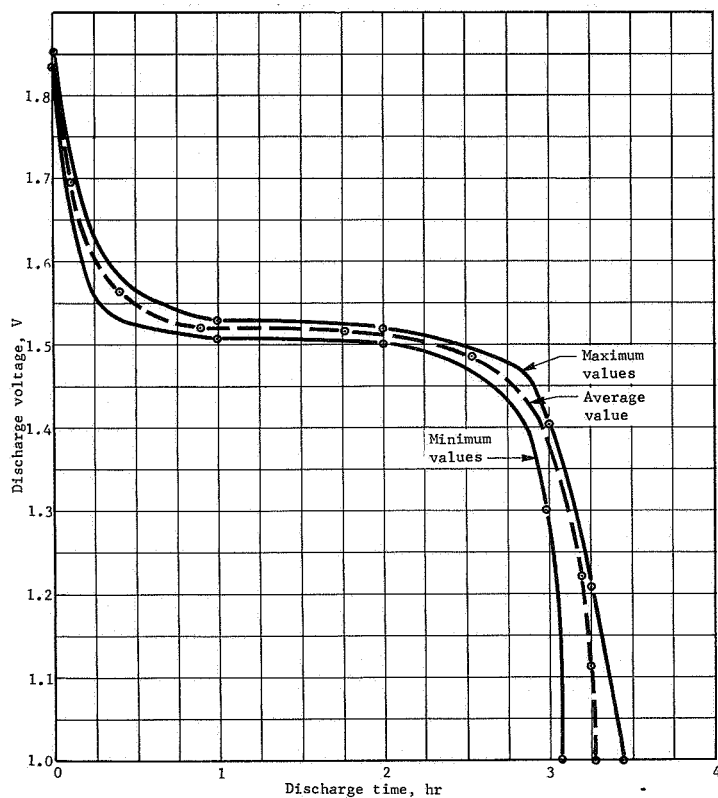


(b) Cycle 3 discharge, cells 37 thru 44

Figure 9.- Sterile Cell Discharge Curves, 10-A Rate  
(125°C Cellophane Activated Cells)



(c) Cycle 4 discharge, cells 37 thru 44



(d) Cycle 5 discharge, cells 37 thru 44

Figure 9.- Concluded

TABLE 9.- STERILE (125°C CELLOPHANE) CELL GROUPS CHARGE AND DISCHARGE AMPERE-HOURS

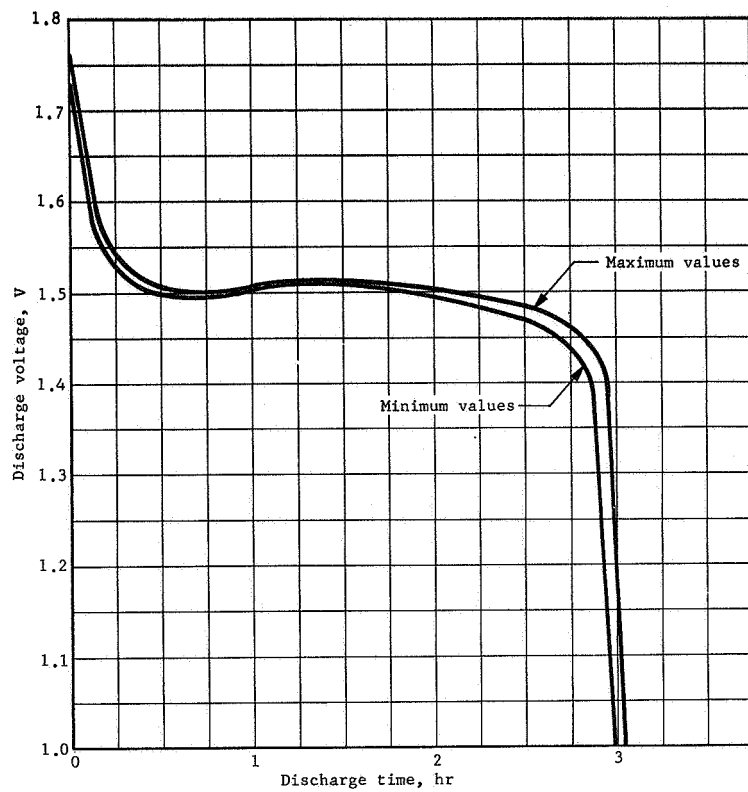
Cell	Cycle 1 <sup>a</sup>			Cycle 2			Cycle 3			Cycle 4			Cycle 5			Cell average		
	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %	Charge	Dis-charge	Effi- ciency, %
37	37.2	18.8	50.5	31.2	<sup>a</sup> 5.0	<sup>a</sup>	34.8	33.5	96.3	33.9	33.2	97.9	35.0	34.5	98.6	33.7	35.7	100.0
38	35.9	24.0	66.9	27.5	24.0	87.3	33.0	32.7	99.0	33.6	32.0	95.2	33.2	32.8	98.8	31.8	30.4	95.6
39	35.4	24.5	69.2	25.5	22.6	88.6	33.1	32.7	98.8	33.3	32.8	98.5	31.9	30.8	96.6	31.0	29.7	95.8
40	37.2	22.8	61.3	31.2	18.8	60.3	34.0	33.0	97.3	33.8	32.8	97.0	33.4	32.8	98.2	33.1	29.4	88.8
41	33.0	27.8	84.2	28.5	27.2	95.4	33.9	33.0	97.3	33.6	33.0	98.2	33.4	32.7	97.9	32.4	31.5	97.2
42	35.8	27.8	77.7	29.1	28.1	96.6	34.0	33.2	97.6	33.9	32.8	96.8	34.0	32.8	96.5	32.8	31.7	96.6
43	34.6	29.0	83.8	29.1	28.1	96.6	33.5	33.0	98.5	33.6	33.0	98.2	33.2	32.7	98.5	32.4	31.7	97.8
44	35.7	24.5	68.6	29.1	27.8	95.5	33.3	32.8	98.5	33.0	32.7	99.1	33.1	32.5	98.2	32.1	31.5	98.1
Cycle aver- age	35.6	24.9	69.9	28.6	25.2	88.1	33.7	33.0	97.9	33.6	32.8	97.6	33.4	32.7	97.9			
Cell group aver- age																32.4	31.2	96.3
45	34.7	31.1	89.6	31.4	31.2	99.4	31.3	30.0	95.8	30.6	31.2	<sup>a</sup> 101.9	32.3	30.5	94.4	31.4	30.7	97.8
46	34.0	30.0	88.2	29.9	29.5	98.7	29.6	28.8	97.3	29.1	29.3	<sup>a</sup> 100.7	29.0	28.3	97.6	29.4	29.0	98.6
47	34.5	31.1	90.1	31.1	30.8	99.0	30.5	29.7	97.4	30.2	30.3	<sup>a</sup> 100.3	30.2	29.8	98.7	30.5	30.2	99.0
48	33.6	29.3	87.2	29.6	29.2	98.6	28.8	28.8	100.0	28.6	28.3	<sup>a</sup> 100.7	28.3	27.5	97.2	28.8	28.5	99.0
Cycle aver- age	34.2	30.3	88.6	30.5	30.1	98.7	30.0	29.3	97.7	29.6	29.9	<sup>a</sup> 100.9	29.9	29.0	97.0			
Cell group aver- age																30.0	29.6	98.7

<sup>a</sup>Indicates data excluded from average values.

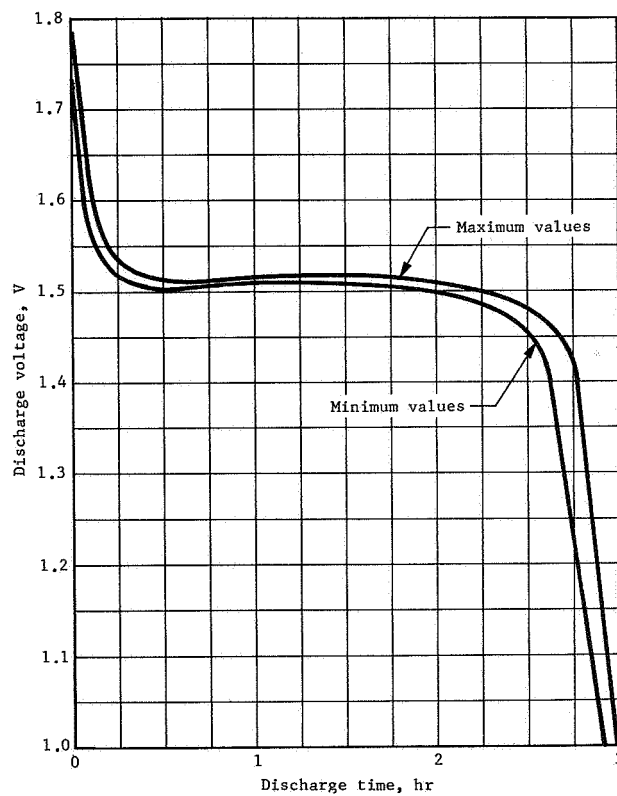
125°C sterile cells activated after 10 weeks dry storage: The cell discharge voltage vs time data for the sterile cells with cellophane sterilized at 125°C and stored 10 weeks before activation (cells 45 thru 48) are shown for cycles 2, 3, 4, and 5 in figure 10. The two solid line curves in this figure indicate the spread in performance during cycles 2, 3, 4, and 5.

The cell voltage during the fifth discharge cycle varied from 1.53 to 1.51 V or a difference of approximately 20 mV during most of the discharge at the Ag<sub>2</sub>O level of the positive electrode before the rapid voltage drop at the end of discharge. The end of discharge time to 1.0 V varied from 2.93 to 1.93 hr or a difference of 1 hr. At the 10-A discharge rate, this represents a 10 A-h spread during a total of 4 discharges. The broken line in figure 10(d) presents the average discharge curve for sterile cells 45 thru 48, indicating that the average Ag<sub>2</sub>O discharge voltage was approximately 1.52 V and the discharge capacity was 26.2 A-h.

The data in lower table 9 summarize the charge and discharge ampere-hours for the stored cells with cellophane sterilized at 125°C. Average values for the ampere-hours accepted on charge and delivered on discharge are shown in the same manner as previously described for the standard and control cell groups. The performance numbers for the stored 125°C group are summarized at the lower right of the table and indicate that the average values for charge and discharge are 30.0 and 29.6 A-h, respectively, with an efficiency of 98.7%. The data obtained during the letdown discharge (cycle 1) at 4 A have been excluded from these averages as previously discussed for the standard and control cell groups.

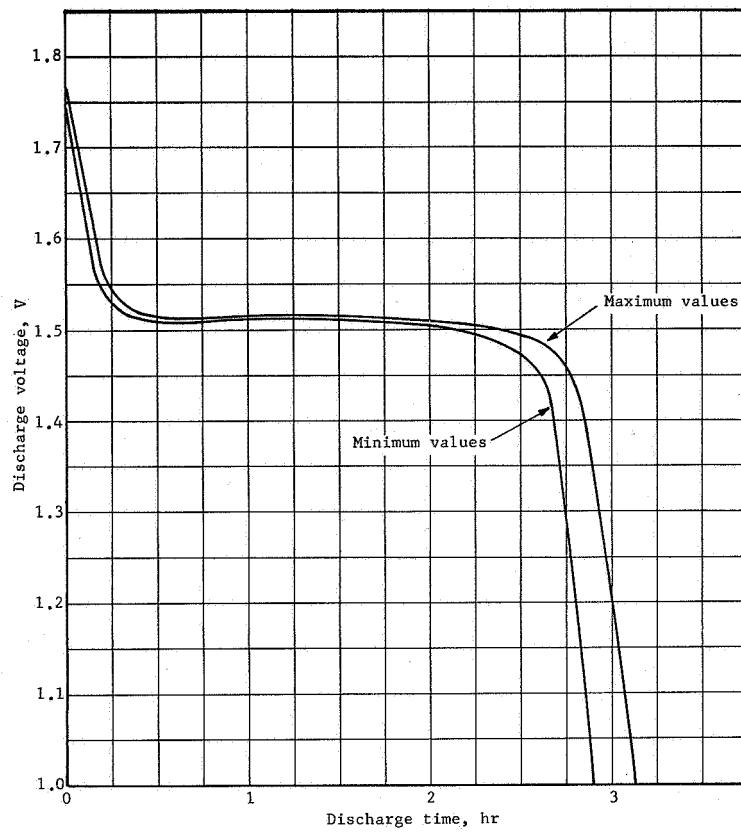


(a) Cycle 2 discharge, cells 37 thru 44

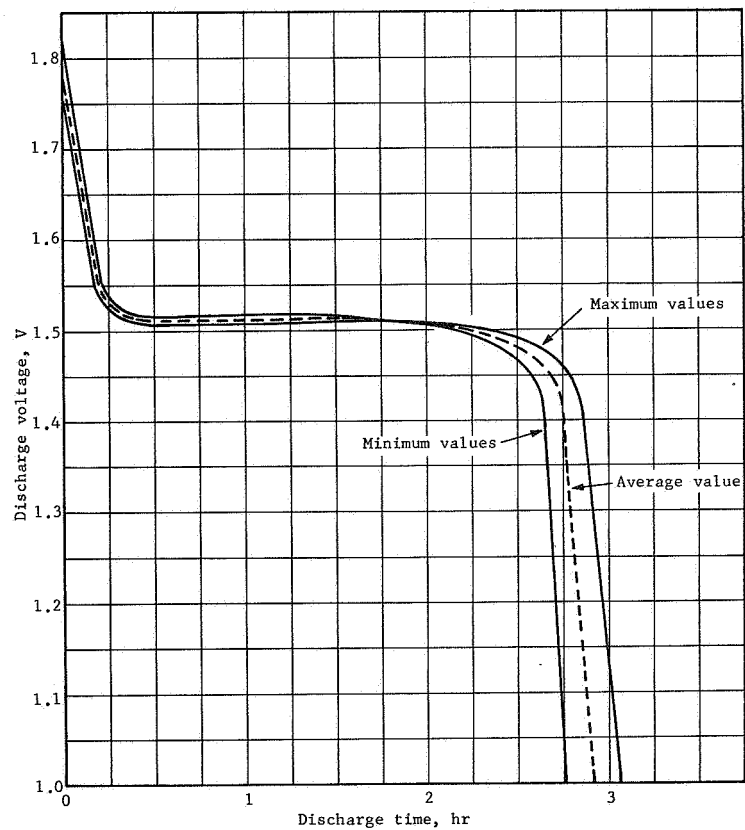


(b) Cycle 3 discharge, cells 37 thru 44

Figure 10.- Sterile Cell Discharge Curves, 10-A Rate  
(125°C Cellophane Stored Cells)



(c) Cycle 4 discharge, cells 37 thru 44



(d) Cycle 5 discharge, cells 37 thru 44

Figure 10.- Concluded



### Cell Performance Comparison

Comparison of sterile cells (cycle 5). - The cell discharge voltage vs time data for the two sterile cell groups with cellophane sterilized at 125°C (cells 37 thru 45 and 45 thru 48) are shown in figure 11 for cycle 5 in comparison with the 135°C cellophane cells. Minimum and maximum values have been shown for the three cell groups by three sets of curves. One set of curves represents the spread of performance of cells using cellophane sterilized at 135°C (cells 25 thru 36). The second set of curves represents the 125°C cells (37 thru 44) which were treated the same as the 135°C cells (25 thru 36), that is, electrolyte was added without a dry stand period. The difference in shortest time to end of discharge to 1.0 V between the 135°C cellophane cells and these activated 125°C cellophane cells is 1.24 hr; the difference in longest time is 0.66. At the 10-A discharge rate these represent 12.4 and 6.6 A-h reductions respectively for the 135°C cellophane cells for cycle 5. Data plotted in the remaining set of curves represent the 125°C cells (45 thru 48) which were dry-stored for 10 weeks prior to addition of electrolyte. The difference in shortest time to end of discharge to 1.0 V between stored cells and the other 125°C cellophane cells is 0.35 hr; the difference in longest time is 0.42. At the 10-A discharge rate these represent 3.5 and 4.2 A-h reductions for the stored-in-dry-condition cells, for cycle 5.

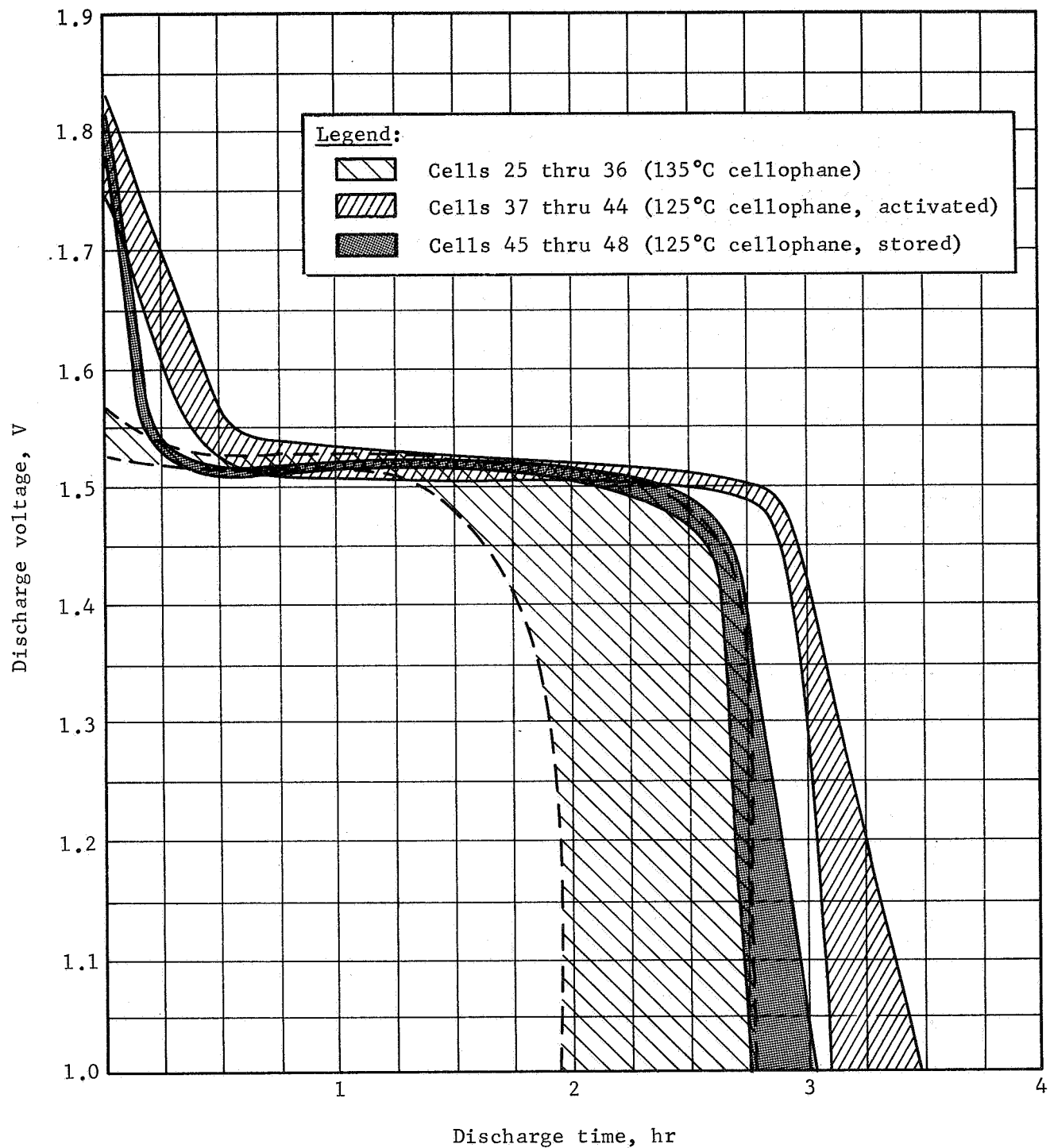


Figure 11.- Discharge Minimum and Maximum Values, Cycle 5

Comparison of cell average performance (cycle 5). - In order to further summarize the discharge characteristics for each cell group, the average data for cycle 5 presented in tables 6 thru 9 is compared in table 10. These data indicate the similarity in performance of the standard, control, and 125°C sterile cell groups on the basis of charge and discharge ampere-hours and efficiency, the effect of sterilizing cellophane at 135°C and the effect of delayed activation. The small difference of delayed activation at 125°C cellophane cells and the larger loss with 135°C cellophane cells is also shown in the table.

TABLE 10. - CELL GROUP AVERAGE DATA COMPARISON FOR CYCLE 5

Performance characteristics	Standard	Control	Sterile		
			135°C cellophane	125°C cellophane activated	125°C cellophane stored
Charge, A-h	33.8	33.8	27.1	33.4	29.9
Discharge, A-h	33.0	33.6	25.1	32.7	29.0
Cycle efficiency, %	97.6	99.2	92.6	97.9	97.0

The average cell discharge voltage vs time data for cycle 5 is shown in figure 12 for the standard, control, and sterile cell groups. These curves indicate the average discharge voltage was essentially the same for all cell groups and that ampere-hours during discharge were essentially the same for the control cell group, the standard cell group, and the sterile cell subgroup with cellophane sterilized at 125°C with immediate electrolyte addition. The sterile cell subgroup with cellophane sterilized at 135°C produced approximately 23% less ampere-hours. The 125°C cells with delayed electrolyte addition showed an 11% loss in capacity when compared with the other 125°C cells.

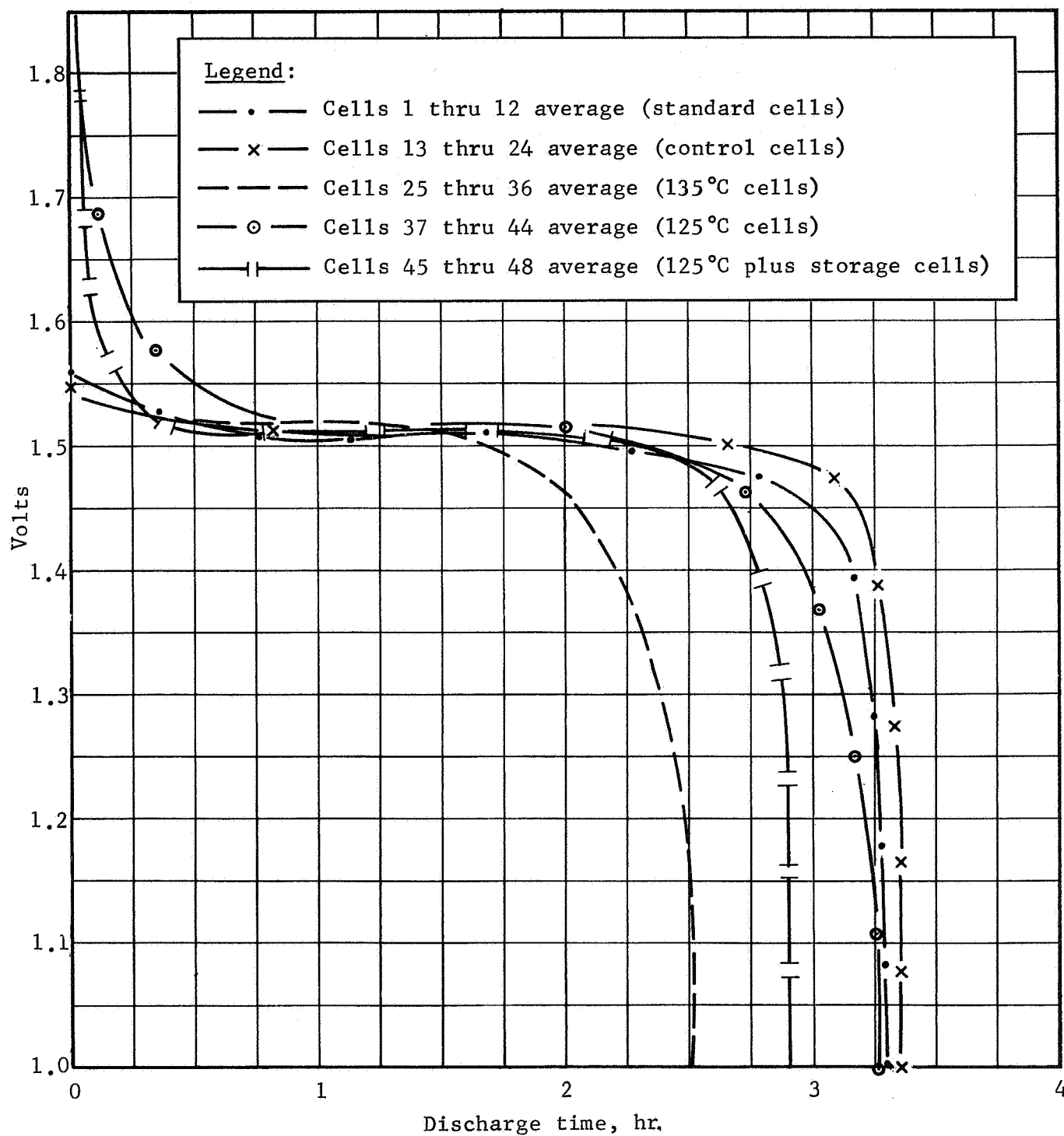


Figure 12.- Average Discharge Comparison, Cycle 5

### Conclusions

- 1) The ESB S-25 silver-zinc cell sterile assembled with 125°C dry heat sterilized cellophane shows no notable change in electrical performance characteristics over five charge/discharge cycles;
- 2) Significant reduction in electrical performance (23% after 5 cycles) occurs with sterile assembled cells using 135°C dry heat sterilized cellophane;
- 3) Minor reduction in electrical performance (11% after 5 cycles) occurs with delayed electrolyte addition to cells assembled with 125°C cellophane;
- 4) Sterile assembly of the ESB S-25 cell appears to be a feasible backup to present investigations to develop a heat sterilizable battery.

## BIOLOGICAL ASPECTS

Sterile assembly of any complex object requires that the entire procedure from piece parts through the finished product be controlled to eliminate the possibility of introducing a single viable organism into the manufacturing process. Implicit in this philosophy is the sterilization of the parts and materials going into the assembly, sterilization, and maintenance of the sterility of the assembly environment and sterility testing of the finished product to verify that the preceding steps did not introduce contamination. In designing a microbiological monitoring and test program, it is necessary to choose among direct and indirect test methods to be employed at given points in the assembly sequence. Direct microbiological assays possess an inherently high risk of contaminating the test article in the very act of performing the assay unless performed under sterile conditions. On the other hand, indirect sterility testing provides information as to the efficacy of the sterilization technique only within the limits and variability of the indicator system. In either case, sterility testing cannot affirm the total absence of organisms; it can only establish that no evidence of contamination has been established in the sample assays. Present practice, therefore, assumes that sterility exists if organisms cannot be recovered by these techniques.

In exploring the feasibility of sterile assembly of an object, the sterility testing procedures must demonstrate that:

- 1) The individual components or parts of the assembly are initially sterile;
- 2) The assembly environment and process contribute no contamination;
- 3) The assembled object is sterile.

It is also necessary that the test methods be universally recognized and accepted as valid to minimize the variability, especially of the indirect assay methods. In the aerospace industry, it has become common practice to employ commercially available spore impregnated strips for indirect sterility testing coupled with the microbiological assay methods described in NHB 5340.1, "NASA Standard Procedures for the Microbiological Examination of Space Hardware." The sterility testing methods employed during this program were either directly taken from these NASA procedures or were slightly modified forms of them. These techniques, as used in this program, are described in detail in Appendix E, Microbiological Assay Materials and Methods.

## Preassembly Sterilization Verification

Before starting the actual assembly operations, it was necessary to verify that the various environments to be employed were capable of inducing and maintaining sterility of the components involved in fabricating a sterile battery. A series of experiments was conducted to verify the dry heat and steam sterilization capability of the isolator passclave and the sterility of the isolator assembly environment.

Passclave dry heat cycle. - An initial dry heat cycle of 125°C for 60 hr was chosen and verified by means of indirect assay methods. Typical cell parts and materials were wrapped in aluminum foil along with a spore-dex strip (AMSCO, Erie, Pa.) and placed in the chamber in a thick-walled aluminum box. The chamber was then filled with dry nitrogen and heated to 125°C for 60 hr. At the conclusion of the cycle, the package was transferred into the first isolator chamber, opened, and the spore strip aseptically removed and microbiologically assayed by the method described in Appendix E. Replicate samples of all the parts and materials required for cell assembly showed no contamination. To add further reliability to the cycle, it was decided to preheat the chamber and parts to 125°C for 4 hr before instituting the cycle on parts to be employed in actual cell assembly. The efficacy of this cycle in actual use is shown in table 11.

Autoclave cycle. - Autoclaving (as a backup to dry heat) for assembly tools and materials was established by preparing packages containing the parts and a spore strip located within the package in the most invulnerable position possible with respect to steam penetration. Individual tools were prepared by taping spore strips to the surface. Multiple test runs established the optimum cycle to be the circulation of flowing steam through the chamber for 5 minutes followed by a pressurized steam cycle of 121°C at 21 psi for 30 minutes. The use of this cycle produced sterility in all test and operating cases as shown in tables 12 and 13.

Isolator sterility. - Before the fabrication of the cells, the isolator system was sterilized by means of peracetic acid and ethylene oxide as described in Appendix E. Spore strips were strategically located throughout the system to verify the efficacy of the sterilization procedure. Subsequent assay of the strips indicated total destruction of the spores by the sterilization procedure.

TABLE 11.- HARDWARE FOR STERILE CELL ASSEMBLY

Item	Sterilization	Test method	Results
1. Cell plate wrapping fixture	Autoclave	Spore strip	Negative
2. Spoonula	Autoclave	Spore strip	Negative
3. Spatula, 2	Autoclave	Spore strip	Negative
4. Glass funnel	Autoclave	Spore strip	Negative
5. Soldering fixture	Autoclave	Spore strip	Negative
6. Wire cutters	Autoclave	Spore strip	Negative
7. Cell plate rack (aluminum) <sup>a</sup>	Autoclave	Spore strip	Negative
8. Cell plate rack (stainless steel)	Autoclave	Spore strip	Negative
9. Solder (solid core)	Autoclave	Spore strip	Negative
10. Solder flux	Autoclave	Spore strip	Negative
11. Battery holder (aluminum) <sup>a</sup>	Autoclave	Spore strip	Negative
12. Battery solder (stainless steel)	Autoclave	Spore strip	Negative
13. Instrumentation board	Autoclave	Spore strip	Negative
14. Crescent wrench	Autoclave	Spore strip	Negative
15. Pan, stainless steel, 2	Autoclave	Spore strip	Negative
16. Soldering iron	Autoclave	Spore strip	Negative
17. Jumper wires, 7	Autoclave	Spore strip	Negative
18. Test tube rack	Autoclave	Spore strip	Negative
19. Allen wrench	Autoclave	Spore strip	Negative
20. Scissors, 2	Autoclave	Spore strip	Negative
21. Pencil	Autoclave	Spore strip	Negative
22. Cheese cloth, 20 ft	Autoclave	Spore strip	Negative
23. Cotton pads, 200	Autoclave	Spore strip	Negative
24. Thermometer	Autoclave	Spore strip	Negative
25. Chromatography paper, 22x18 in., 50	Autoclave	Spore strip	Negative
26. Medicine dropper, 4	Autoclave	Spore strip	Negative
27. Nut wrench	Autoclave	Spore strip	Negative
28. Forceps, 2	Autoclave	Spore strip	Negative
29. Tygon tubing, 3/4 and 1/4 in., 5 ft each	Autoclave	Spore strip	Negative
30. Petri dishes, 6	Autoclave	Spore strip	Negative
31. Fuse wire, 3 ft	Autoclave	Spore strip	Negative

<sup>a</sup>Aluminum fixtures were removed after two weeks because of corrosion problems involving the fixture.



TABLE 12.- STERILIZATION OF PARTS FOR CONSTRUCTION  
OF 12 STERILE CELLS (FIRST SET)

Package	Material	Run	Sterilization	Test method	Results
<sup>a</sup> 1	50 negative plates	1	Dry heat	1 spore strip	Negative
2	50 negative plates	1	Dry heat	1 spore strip	Negative
3	50 negative plates	1	Dry heat	1 spore strip	Negative
4	49 negative plates	1	Dry heat	1 spore strip	Negative
5	50 positive plates	1	Dry heat	1 spore strip	Negative
6	50 positive plates	1	Dry heat	1 spore strip	Negative
7	50 positive plates	1	Dry heat	1 spore strip	Negative
8	34 positive plates	1	Dry heat	1 spore strip	Negative
9	26 flat washers and 26 nuts	1	Dry heat	1 spore strip	Negative
10	24 terminal posts	1	Dry heat	1 spore strip	Negative
11	85 pieces of large taffeta and 15 pieces of small taffeta	1	Dry heat	1 spore strip	Negative
<sup>b</sup> 12	12 cases and lids	1	Dry heat	2 spore strips	Negative
<sup>c</sup> 13	100 sheets cellophane	2	Dry heat		
14	24 nuts	3	Autoclave	1 spore strip	Negative
15	4 bottles of 40% KOH	4	Autoclave	1 spore strip	Negative
16	8 bottles of 40% KOH	5	Autoclave	2 spore strips	Negative

<sup>a</sup>Packages 1 to 11 were wrapped first in chromatography paper, then in aluminum foil. A spore strip was located in the center of the packet. Packages 1 to 11 were heated in an aluminum box in the sterilization chamber.

<sup>b</sup>Cases and lids were not wrapped. A spore strip was taped to the exterior of one case and to the interior of another.

<sup>c</sup>The cellophane was rolled onto a glass drum and placed inside a closed jar. Spore strips were located at the interior of the roll and on the exterior. The cellophane was sterilized at 135°C for 60 hr.

TABLE 13.- STERILIZATION OF PARTS FOR CONSTRUCTION  
OF 12 STERILE CELLS (SECOND SET)

Package	Material	Run	Sterilization	Test method	Results
<sup>a</sup> 1	50 negative plates	1	Dry heat	1 spore strip	Negative
2	50 negative plates	1	Dry heat	1 spore strip	Negative
3	50 negative plates	1	Dry heat	1 spore strip	Negative
4	49 negative plates	1	Dry heat	1 spore strip	Negative
5	50 positive plates	1	Dry heat	1 spore strip	Negative
6	50 positive plates	1	Dry heat	1 spore strip	Negative
7	50 positive plates	1	Dry heat	1 spore strip	Negative
8	34 positive plates	1	Dry heat	1 spore strip	Negative
9	24 flat washers and 24 nuts	1	Dry heat	1 spore strip	Negative
10	24 terminal posts	1	Dry heat	1 spore strip	Negative
11	85 pieces of large taffeta	1	Dry heat	2 spore strips	Negative
12	15 pieces of small taffeta	1	Dry heat	1 spore strip	Negative
<sup>b</sup> 13	100 sheets cellophane	2	Dry heat	2 spore strips	Negative
<sup>c</sup> 14	24 nuts	3	Autoclave	1 spore strip	Negative
15	12 bottles of 40% KOH	4	Autoclave	3 spore strips	Negative
16	12 cases and lids	5	Autoclave	2 spore strips	Negative
<sup>a</sup> Packages 1 to 12 were wrapped first in chromatography paper, then in aluminum foil. A spore strip was placed in the center of each packet. Packet 11 had two spore strips. All packets were placed directly in the heating chamber. No aluminum box was used.					
<sup>b</sup> Cellophane was sterilized the same as in set one.					
<sup>c</sup> The nuts were autoclaved in a petri dish along with a spore strip.					

## Assembly Sequence Microbiological Monitoring and Control

General controls. - Although the experiments described previously had verified the efficacy of the sterilization processes and the assembly environment, it was deemed necessary to institute additional precautions and controls during cell assembly to minimize the possibility of the introduction of accidental contamination. Among the general controls employed were the use of a three-isolator system and the inclusion of ultraviolet lamps in the isolator chambers.

The isolator system, described in detail in other sections of this report, consisted of three chambers interconnected by double-door passthroughs. During cell assembly operations each of the three chambers was used for a specific purpose. The first chamber, opened into by the sterilization chamber, was used as a sterile storage area. In this chamber, packages of parts and materials that had undergone sterilization were held until biological assay of the indicator spore strips included in the packages verified their sterility. As needed, the parts and materials were transferred to the second isolator, in which actual assembly operations were performed. The completed cells were then passed into the third chamber where electrical testing was performed. In this way, any accidental contamination of one of the chambers could be prevented from affecting the entire system.

A further precaution was instituted by the inclusion of ultraviolet lamps in all three chambers. Although ultraviolet light is of low efficiency in sterilizing large volumes of moving air, it was felt that, in the static atmosphere of the chambers, the maintenance of sterility of the chambers would be enhanced by their use.

Specific controls. - Specific controls are discussed in the following paragraphs.

Atmosphere monitoring: Throughout the assembly operations, the atmospheres within the isolators were monitored. This was accomplished by employing 0.45- $\mu$  field monitors (Millipore Corp.) on all the inlet/outlet valves of the isolator system (fig. 13). These filters were removed and biologically assayed at the conclusion of the assembly operations. No contamination was detected.

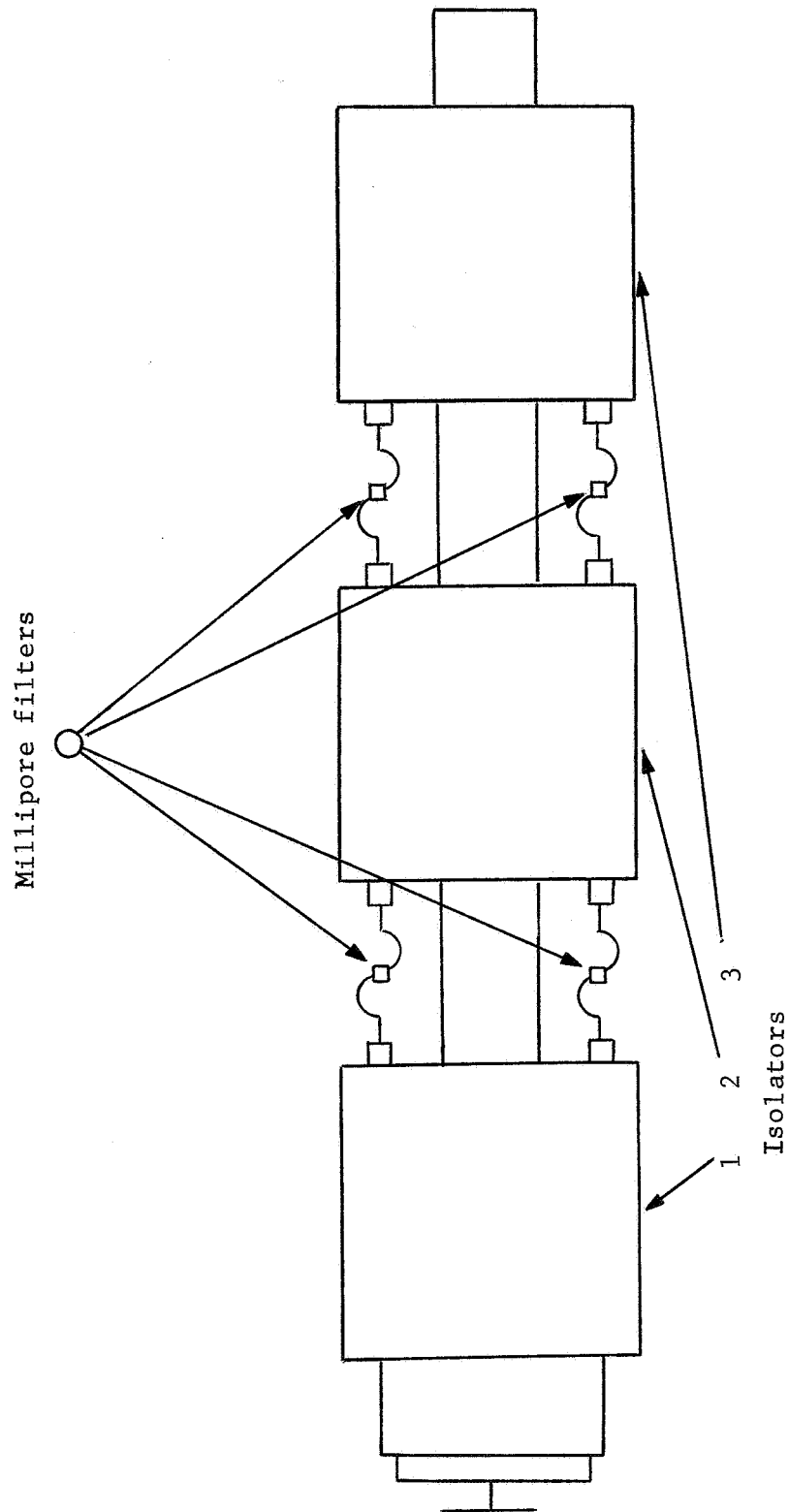


Figure 13.- Isolator Airborne Contamination Monitoring System

Trypticase Soy Agar settling plates were employed in the isolators and passthroughs. These petri dishes were replaced at weekly intervals and incubated. No contamination was detected during the assembly program.

Glove assays: The possibility of an undetected puncture of the isolator glove system was identified as a potential contamination source. To preclude this possibility, weekly impressions were taken of the finger tips of the gloves. The fingers of the gloves were impressed into TSA plates placed in the isolators. These were then replaced, incubated, and examined for growth. No contamination was detected during the assembly program.

Isolator surface assays: During the final two weeks of assembly operations, two sets per week of swab assays were made of interior surfaces of the isolators. Each set of assays consisted of 14 individual samples. No contamination was detected.

Summary. - Accepted, standard microbiological techniques (described in detail in Appendix E) were employed throughout the assembly program to monitor all potential sources of microbial contamination. The method employed in each case was selected on the basis of:

- 1) The physical characteristics of the situation to be monitored;
- 2) The acceptability of the method to the scientific community;
- 3) The sensitivity and reliability of the method;
- 4) The practicality of its application to the situation in question.

Of the many replicate samples taken and assays run, not a single case of contamination was detected. These tests included the indirect testing of the sterility of the cell parts and materials introduced into the isolators and the direct testing of the assembly environment.

## Post-Electrical Test Microbiological Assay

Although the microbiological monitoring conducted during the assembly operation demonstrated the sterility of the cell components, the assembly environment and the tools and materials employed, the final verification of cell sterility can only be made by demonstrating that the completed cell is indeed sterile. This demonstration requires disassembly of the cell and microbiologically assaying the components. To maintain the balanced experimental design in the electrical performance tests, disassembly to the component level was accomplished after the completion of the charge/discharge cycles. Concurrently, this disassembly permitted examination of the interior cell components to ascertain the changes occurring from the electrical operations.

The assay of disassembled components required a basic change in the microbiological procedures that introduced a higher risk of contamination during assay. Removal of organisms from the surface of the components necessitates a procedural step for insonation in peptone water, and plating of the peptone supernatant liquid so that adverse effects (i.e., possibly inhibiting growth) of the components does not bias the results. To conduct the insonation operation inside the isolator requires sterilizing the instrument in the passclave before entry into the isolator. This approach did not appear to be suitable because:

- 1) Compatibility information was not available on sterilizing the ultrasonicator with dry heat or by autoclave means. To obtain such data would require an extensive program outside the scope and time period of the contract;
- 2) Use of an arbitrarily selected sterilization method could result in damage to the instrument necessitating costly and time-consuming repairs or replacement. This risk appeared to be unacceptable in the light of the program schedule.

It was therefore decided to conduct these assay procedures external to the isolator system recognizing the inherent risks associated with the choice.

Results of the assays performed on the cells assembled under sterile conditions are shown in tables 14 thru 16. Contamination was found in cells 26, 27, 28, and 33. All other cells examined showed no contamination. The four contaminations encountered are believed to have been introduced during the assay procedures. The bases for this belief are discussed below.

TABLE 14.- BIOLOGICAL ASSAY OF STERILE ASSEMBLED CELLS,  
BATTERY 1

Cell	Cell part	Results	Cell	Cell part	Results
25	Nuts, washers, cap	Negative	27	Nuts, washers, cap	Negative
25	Terminals	Negative	27	Terminals	Negative
25	Negative plate	Negative	27	Negative plate	Negative
25	Negative plate	Negative	27	Negative plate	Negative
25	Cellophane	Negative	27	Cellophane	Negative
25	Positive plate	Negative	27	Positive plate	Negative
25	Positive plate	Negative	<sup>b</sup> 27	Positive plate	Negative
25	Taffeta	Negative	27	Taffeta	Negative
26	Nuts, washers, cap	Negative	28	Nuts, washers, cap	Negative
26	Terminals	Negative	<sup>c</sup> 28	Terminals	Positive
26	Negative plate	Negative	28	Negative plate	Negative
26	Negative plate	Negative	28	Negative plate	Negative
26	Cellophane	Negative	28	Cellophane	Negative
26	Positive plate	Negative	28	Positive plate	Negative
<sup>a</sup> 26	Positive plate	Positive	28	Positive plate	Negative
26	Taffeta	Negative	28	Taffeta	Negative
<sup>a</sup> Contamination was <u>B. subtilis</u> - gram positive rods. <sup>b</sup> Contamination was <u>B. subtilis</u> - gram positive rods. <sup>c</sup> Contamination appeared to be <u>E. coli</u> - gram negative rods.					

TABLE 15.- BIOLOGICAL ASSAY OF STERILE ASSEMBLED CELLS,  
BATTERY 2

Cell	Cell part	Results	Cell	Cell part	Results
29	Nuts, washers, cap	Negative	31	Nuts, washers, cap	Negative
29	Terminals	Negative	31	Terminals	Negative
29	Negative plate	Negative	31	Negative plate	Negative
29	Negative plate	Negative	31	Negative plate	Negative
29	Cellophane	Negative	31	Cellophane	Negative
29	Positive plate	Negative	31	Positive plate	Negative
29	Positive plate	Negative	31	Positive plate	Negative
29	Taffeta	Negative	31	Taffeta	Negative
30	Nuts, washers, cap	Negative	32	Nuts, washers, cap	Negative
30	Terminals	Negative	32	Terminals	Negative
30	Negative plate	Negative	32	Negative plate	Negative
30	Negative plate	Negative	32	Negative plate	Negative
30	Cellophane	Negative	32	Cellophane	Negative
30	Positive plate	Negative	32	Positive plate	Negative
30	Positive plate	Negative	32	Positive plate	Negative
30	Taffeta	Negative	32	Taffeta	Negative



TABLE 16.- BIOLOGICAL ASSAY OF STERILE ASSEMBLED CELLS,  
BATTERY 3

Cell	Cell part	Results	Cell	Cell part	Results
33	Nuts, washers, cap	Negative	35	Nuts, washers, cap	Negative
33	Terminals	Negative	35	Terminals	Negative
33	Negative plate	Negative	35	Negative plate	Negative
33	Negative plate	Negative	35	Negative plate	Negative
33	Cellophane	Negative	35	Cellophane	Negative
33	Positive plate	Negative	35	Positive plate	Negative
33	Positive plate	Negative	35	Positive plate	Negative
<sup>a</sup> 33	Taffeta	Positive	35	Taffeta	Negative
34	Nuts, washers, cap	Negative	36	Nuts, washers, cap	Negative
34	Terminals	Negative	36	Terminals	Negative
34	Negative plate	Negative	36	Negative plate	Negative
34	Negative plate	Negative	36	Negative plate	Negative
34	Cellophane	Negative	36	Cellophane	Negative
34	Positive plate	Negative	36	Positive plate	Negative
34	Positive plate	Negative	36	Positive plate	Negative
34	Taffeta	Negative	36	Taffeta	Negative
<sup>a</sup> Contamination was <u>Staphylococcus</u> sp. - gram positive cocci.					

Cell 26 and 27 positive plate. - The organism isolated in both cases was determined to be Bacillus subtilis (ref. 10). Subsequent Rodac plate assays of the laminar flow bench in which the pour plates were made revealed contamination of the bench with B. subtilis. The bench was decontaminated with 2% peracetic acid before performing further cell assays. In addition, the contaminant was isolated from only one of seven positive plates. Had the initial sterilization of the plates been incomplete, it is unlikely that only one of 84 positive plates sterilized simultaneously would remain contaminated. The likelihood of the contamination being introduced during assembly and surviving through charge/discharge cycling is remote for the following reasons:

- 1) The concentration of silver ions in the electrolyte during operation of the cell would tend to be lethal;
- 2) The increase in KOH temperature during the duty cycle of the cell has been shown to be lethal to B. subtilis spores;
- 3) The positive plate undergoes dissolution and redeposition during cycling of the cell. This would tend to either remove or overlay organisms on the plate surface.

Cell 28 terminals. - The contaminant isolated in this instance was determined to be Escherichia coli, usually associated with fecal contamination. Colony formation was noted only on the surface of the pour plate, and was entirely lacking within the medium. The remaining contents of the bottle from which the pour plate was made evidenced no bacterial contamination. It should also be noted that during soldering of the cell terminals and during cell operations the terminals reach a temperature sufficiently high as to be considered lethal for this organism.

Cell 33 taffeta. - The organism involved in this contamination incident was identified as a staphylococcus, another organism usually considered indicative of contamination from a human source. Cell components adjacent to and in direct contact with the taffeta showed no contamination. This fact, coupled with the severity of the internal cell environment discussed above, seems to point to the introduction of the contaminant during the assay procedure.

## Conclusions

The results of this study have established the feasibility of sterile assembly of batteries. This conclusion is based on the conviction that the four contamination cases that appeared were the result of technical error in conducting the microbiological assays and not due to a failure in the sterile assembly process. It should be further noted that future sterile assembly situations must allow for conduct of the microbiological sample handling entirely within the isolator system. This approach would most certainly preclude the introduction of accidental contamination experienced in this program.

## PACKAGING CONCEPTS

After sterile assembly of a battery, all subsequent operations in the total factory-through-launch sequence must assure maintenance of the sterile condition. Packaging concepts are therefore needed that recognize such factors as:

- 1) Assembly of the packaging inside the isolator - This requirement restricts usage of packaging materials to those compatible with passclave sterilization;
- 2) Environmental testing - Flight acceptance environmental testing is a program requirement for all flight hardware. Packaging designs must, therefore, permit reasonable reproduction of the critical flight environments on the packaged item as well as performance type measurements while in the package;
- 3) Biological security during transportation - Packaging for shipment to the launch site must maintain biological security (sterility) under the rough treatment encountered in transportation;
- 4) Assembly sterilizer and sterile-insertion-through-port opening interface - Launch site operations will entail removal of the sterile battery from the package. Therefore, the packaging designs must recognize the interface with these operations.

Brainstorming sessions generated a large number of packaging schemes. Upon further evaluation, six concepts were identified as having basic merit. These six concepts are described in the ensuing subsections of this section. These concepts were reviewed with NASA-Langley personnel as part of the technical review meetings. The reviews led to selection of two concepts (split seam package and flap-type sterile seam) as warranting further consideration.

### Hermetically Sealed Stainless Steel Case

This concept (fig. 14) is based on (1) the hermetic seal to maintain internal sterility, and (2) flash heating, chemical sterilization, or a similar surface sterilization method to cleanse the outer surfaces. This concept has a primary advantage of simplicity; its disadvantages are additional weight for the stainless steel container; nonavailability today of acceptable surface sterilization treatments; and its applicability to batteries, but not to the universal class of possible heat-sensitive items.

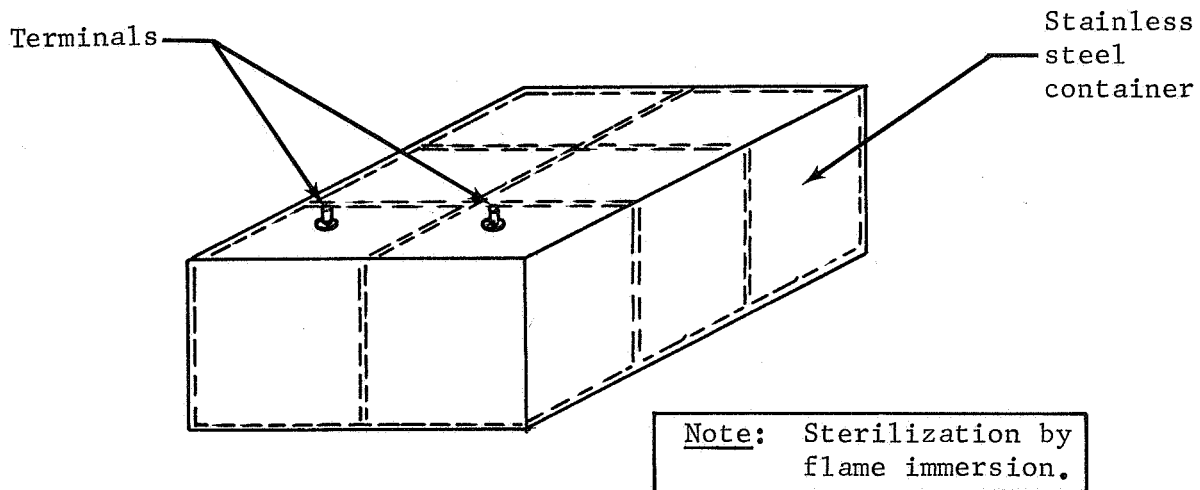


Figure 14.- Hermetically Sealed Stainless Steel Battery Case Concept

### Thermal Barrier Package

This concept (fig. 15) uses a double wall container, filled with insulation, for packaging. Rigid supports for the battery adds an air pocket as an additional insulating medium. The exterior contaminated surface of the package can be sterilized by a dry heat cycle that will assure surface kill of contamination with only slight internal temperature rise. Rigidity and weight associated with this concept precludes its use for environmental testing.

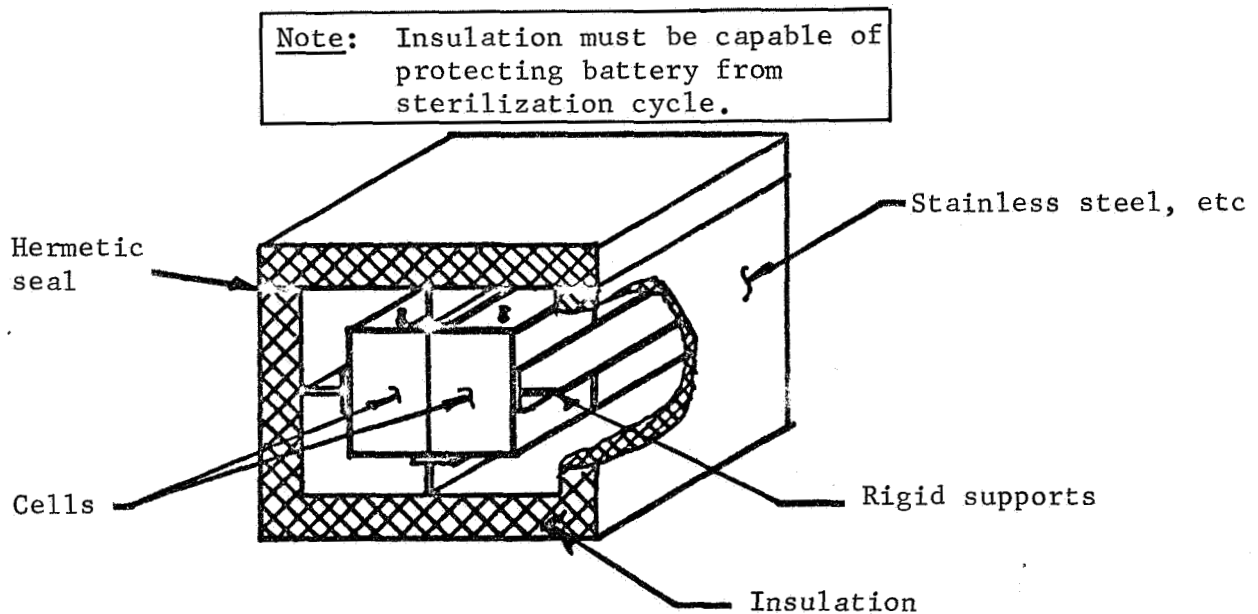


Figure 15.- Thermal Barrier Ship and Test Package Concepts

### Heat Exchanger Package

This concept (fig. 16) uses the chemical flux principle to keep the internal battery components cool during a dry heat sterilization cycle. The normal battery case is replaced by a double-walled container with inlet and outlet openings to permit circulation of water or other liquid cooling media. Insulation on the interior of the outer wall permits adequate heating to effect biological kill on the surface. This substitution of battery case materials and design would require a development program and qualification testing.

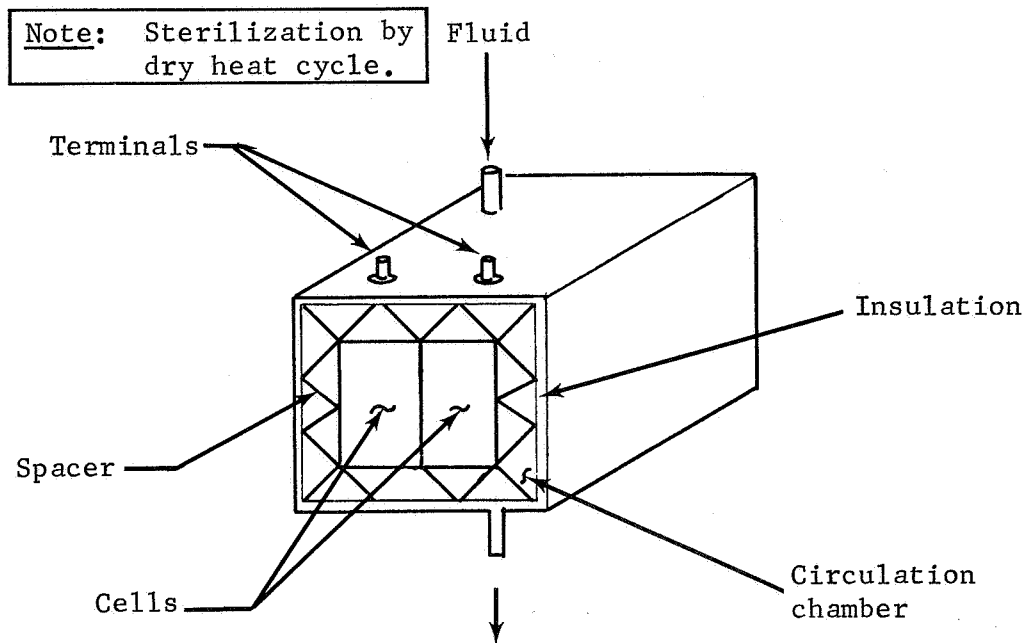


Figure 16.- Heat Exchanger Package Concept

### Shipping Container Service Chamber

This concept (fig. 17) uses a modification of the sterile-insertion concepts developed under Contract NASw-1621. It therefore interfaces with the sterile-insertion-through-port openings technique. However, structural rigidity associated with the design would affect the data obtained during flight acceptance testing.

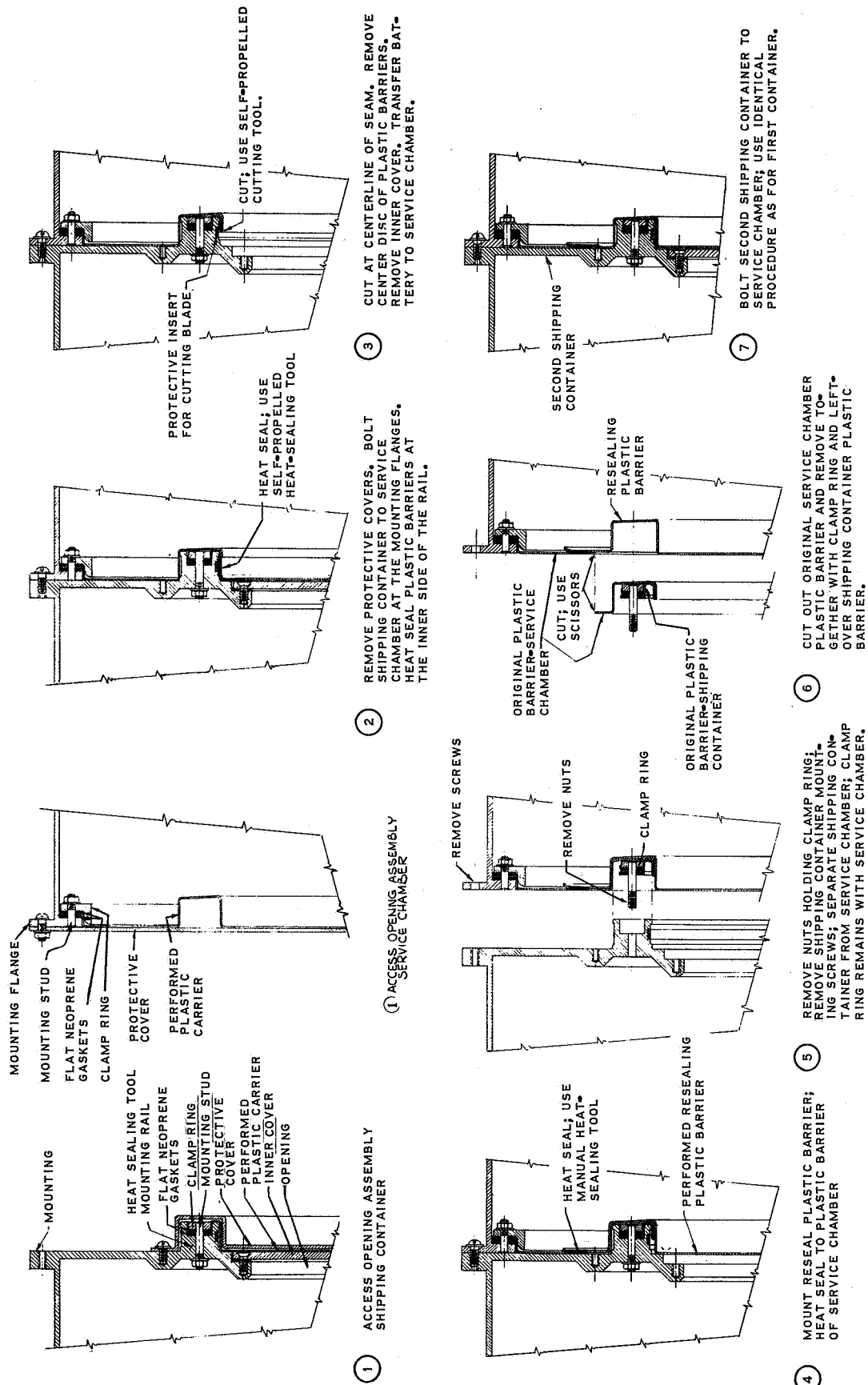


Figure 17.- Shipping Container Service Chamber Concept



### Split Seam Package

This concept (fig. 18) is compatible with techniques proven under NASA Contracts NASw-1407 (ref. 6) and NAS8-21122 (ref. 8), which identified the feasibility of plastic films for sterile insertion. The concept uses a non-heat-sealable material in the sterile package access sleeve, which inhibits a seal between the two inner surfaces, during attachment of a transfer sleeve, with the heat sealer/cutter. Featured in the concept is a mechanically mounted sealing device located on the inner wall of the transfer chamber (fig. 19). The pantograph linkage of the sealing device mount assures a parallel and uniform pressure of the device over the entire seam area. The device may be designed for manual operation or remotely driven by electromechanical, pneumatic, or other means. This concept permits numerous transfers, because each transfer requires only a couple of inches of the transfer sleeve. The sleeve can be replaced when required. In view of its many desirable features, this concept has been selected for possible future development.

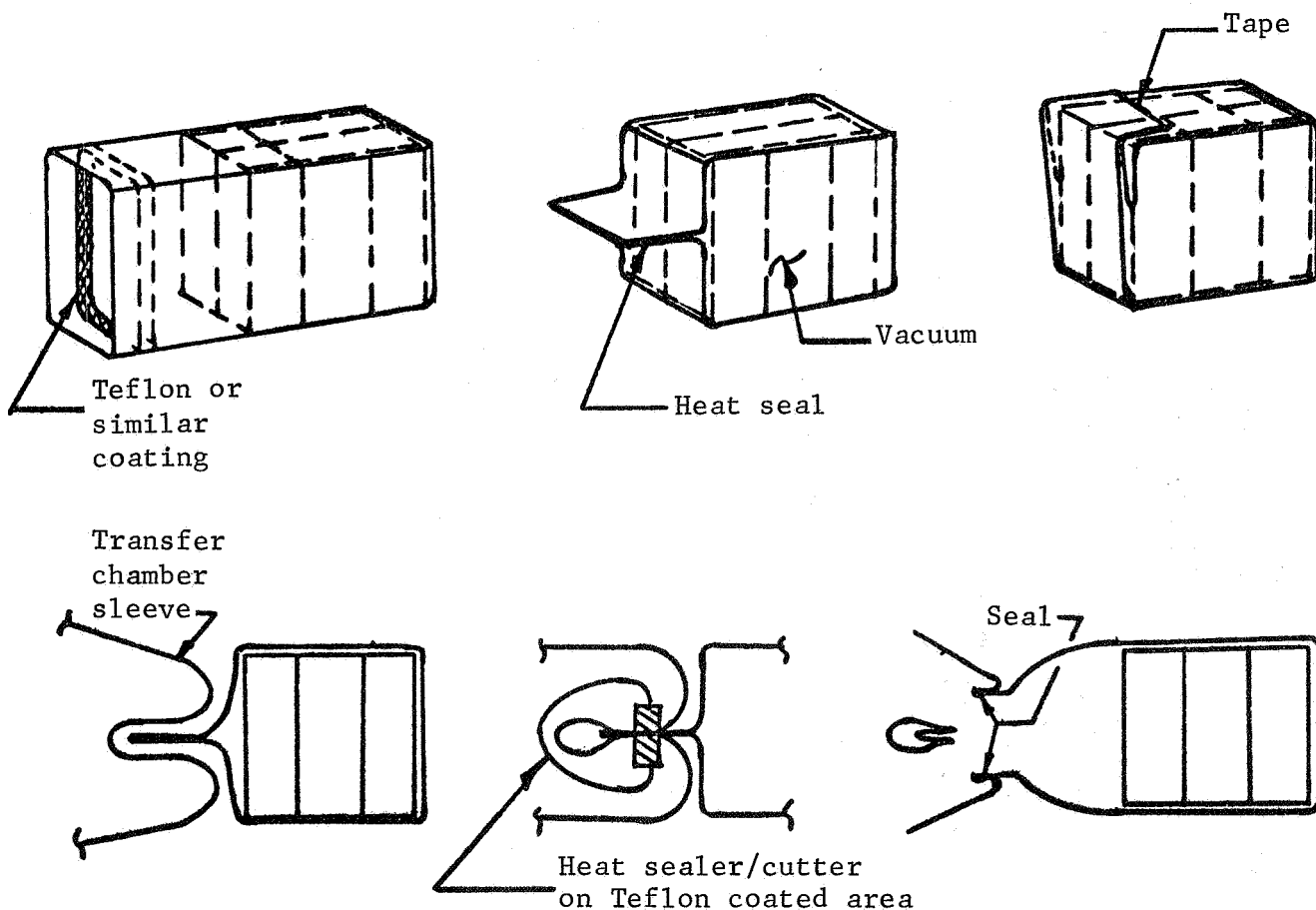


Figure 18.- Split Seam Insertion Technique

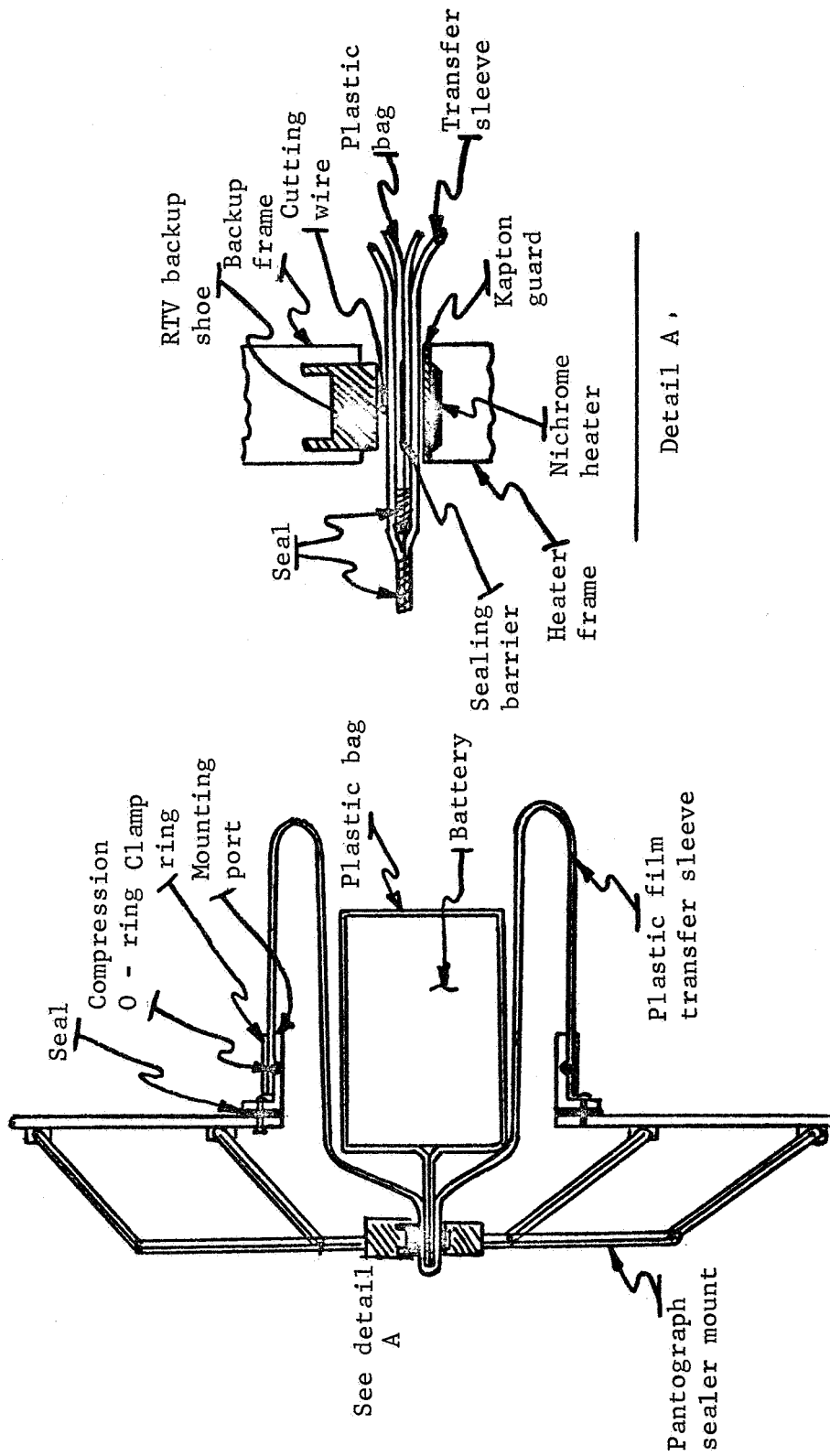


Figure 19.- Split Seam Packaging Concept

### Flap-Type Sterile Seam

This concept (fig. 20) is also an offshoot of design concepts developed under Contract NASw-1621. The battery is firmly held in a frame that is attached to a baseplate. This baseplate serves as a mounting fixture for spacecraft assembly. Concurrently, it is designed to serve as a heat sink, backup ring for a rectangular sealing tool. The opening is covered by a plastic film barrier. The baseplate has a large dimension slightly greater than the width of the battery case and a smaller dimension slightly larger than the height of the battery. Using a correspondingly shaped heat sealing tool, plastic-to-plastic sealing is accomplished against the baseplate following the technology of the NASw-1621 contract. After cutting through the seam area, the battery is passed through the opening. This concept will require either standardization of the interface with the removal operations at the launch site or separate heat-sealing tools to match particular package designs. This concept has also been selected for possible future development.

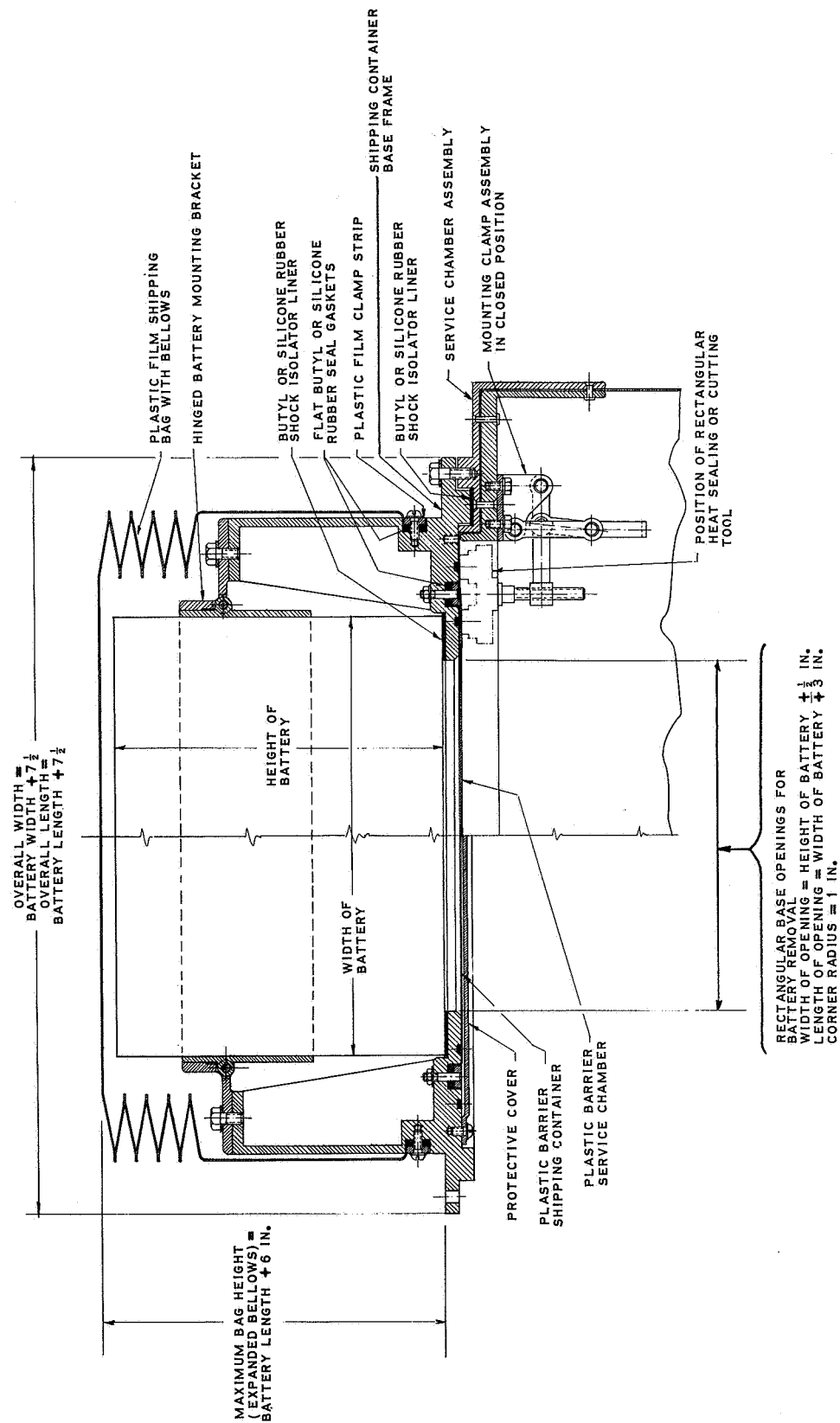


Figure 20.- Flap-Type Sterile Seam Concept

## CONCLUSIONS AND RECOMMENDATIONS

### Conclusions

The following conclusions can be drawn from this investigation:

- 1) Sterile Assembly of ESB S-25 silver-zinc cells from heat sterilized components is feasible. This conclusion stems from the following cogent points.
  - a) No major discomfort or loss of manual dexterity needed for assembly was observed or reported by the operators.
  - b) A definitive time and motion study was not conducted as part of the program, so a direct measure of assembly time is not available. However, conformance to the schedule as a measure of assembly time is indicative of no appreciable difference in assembly time. (This statement, of course, excludes the additional time required for isolator decontamination, and passclave operations for materials and equipment.)
  - c) Use of a multiple isolator system permits concurrent assembly operations, electrical testing, and biological controls. This arrangement appears to be adaptable, in principle, to an assembly line operation. In addition, isolation of each isolator yields a safeguard against inadvertent contamination of the total operation if a breach of biological security occurs in one operation.
  - d) Although four contaminated assays were obtained on the disassembled components after electrical testing, these are believed to stem from technician error for the reasons cited in the section entitled BIOLOGICAL ASPECTS. This condition points up to the mandatory nature of conducting biological control assays under totally sterile conditions -- preferably inside the isolator system including the final incubation. We feel it would be an error to conclude that these data invalidate the feasibility demonstration.

- e) Personnel were indoctrinated on the importance of strict adherence to procedures to avoid potential breaches of security or malassembly. No malfeasance was noted during the study, thus lending credence to an ability to maintain similar discipline by battery manufacturer assembly personnel.
- 2) Sterile assembly of ESB S-25 silver-zinc cells provides a potentially acceptable backup to present investigations on development of a sterilizable battery. This conclusion can be drawn from the following.
- a) The charge/discharge data for the sterile assembled cells with cellophane sterilized at 125°C differs only slightly from the ampere-hour capacity and charge efficiency of the standard and control cells.
  - b) Sterile assembly of batteries required additional controls and special handling that would not be required if a sterilizable battery were developed. In view of these extra requirements, only a back-up status is warranted.
  - c) Unknown degradation effects may still be present and become apparent in environmental (qualification/flight acceptance testing) or life testing of the cells.
- 3) Suitable packaging concepts are now available for maintaining sterility of sterile assembled hardware in the factory-through-launch sequence. This conclusion stems from the two preferred concepts.
- a) The split seam package allows reasonable reproduction of environmental stresses during flight acceptance testing and can be easily adapted to performance measurements. Additional protective packaging may be required during transit. Removal concepts are known to be feasible from earlier contract work.
  - b) The flap-type sterile seam also meets these criteria. Note, however, that the mounting bracket in the spacecraft should correspond to the baseplate design so that vibration tests of the battery on the baseplate would be reasonably representative.

### Recommendations

The following recommendations can be drawn from this investigation:

- 1) Development and evaluation of sterile packaging concepts should be instituted. Hardware investigations are now underway in MAST and the sterile-insertion-through-port-opening technique. This study has shown the feasibility of sterile assembly of batteries and, by inference, sterile assembly of heat-sensitive items. To complete the total system for use on future missions, suitable packaging must be developed and evaluated.
- 2) A qualification/life test program should be considered for sterile assembled batteries. This investigation only demonstrated the feasibility of sterile assembly of ESB S-25 silver-zinc cells with no significant electrical degradation after five charge/discharge cycles. Additional evaluation of silver-zinc cells assembled as batteries is required before mission application could be recommended. A qualification program, limited to critical environments, appears to be desirable with a life test after the exposures to determine unknown electrical degradation effects from the environments and the sterile assembly. The life test could also include sample cell removal at periodic intervals, disassembly, and examination of deposition on the separators for possible use as an accelerated life test method.

Martin Marietta Corporation  
Denver, Colorado, July 3, 1968

## APPENDIX A

### EQUIPMENT DESCRIPTION

#### Environmental Control Equipment

The following is a description of materials and components used in the isolator system.

Sterilization oven (passclave). - The stainless steel pass-clave (fig. A1) can be used to provide moist heat at 121°C or dry heat sterilization. It has sealed covers on the front and back and is bolted and sealed to the isolator system. An automatic temperature controller, a temperature indicator, and inlet and vent valves are mounted in the top and bottom. (Mfg: Kewaunee Scientific Equipment, Order Number 718-SS.)

Controlled atmosphere chambers (isolators). - The three isolators have an overall dimension of 13 ft 3 1/8 in. by 43 in. by 25 in. The isolators are joined by interchange compartments, 12x12x12 in., which have sealed doors on both ends and inlet/vent valves on the top and bottom. Four glove ports equipped with neoprene gloves (Charleston Rubber Co.) are mounted in each isolator (two ports per isolator side). Each chamber is also equipped with inlet/vent valves (two per isolator side). An ultraviolet lamp is mounted inside each chamber of the isolator system. The isolator setup is shown in figure A2. (Mfg: Kewaunee Scientific Equipment, Model 2C391.)

Electro-hygrometer. - The hygrometer sensing unit is mounted in the roof of chamber 1. The hygrometer was used to measure humidity from 0 to 100%. (Mfg: Lab-Line Instrument Company.)

Additional environmental support items. - The following is a list of materials used as an adjunct to the basic isolator system (an explanation of the respective use of the items listed will be found in the body of the report):

- 1) Aluminum foil (Alcoa);
- 2) Chromatography paper (Whatman);
- 3) Anaerobic jar (BBL);
- 4) Nitrogen (Linde);
- 5) Petri dishes (Van Waters and Rogers);
- 6) Flask (Pyrex, 2 liter);
- 7) Tygon tubing (1/4 in. inside diameter);
- 8) Displacement bladder.



APPENDIX A

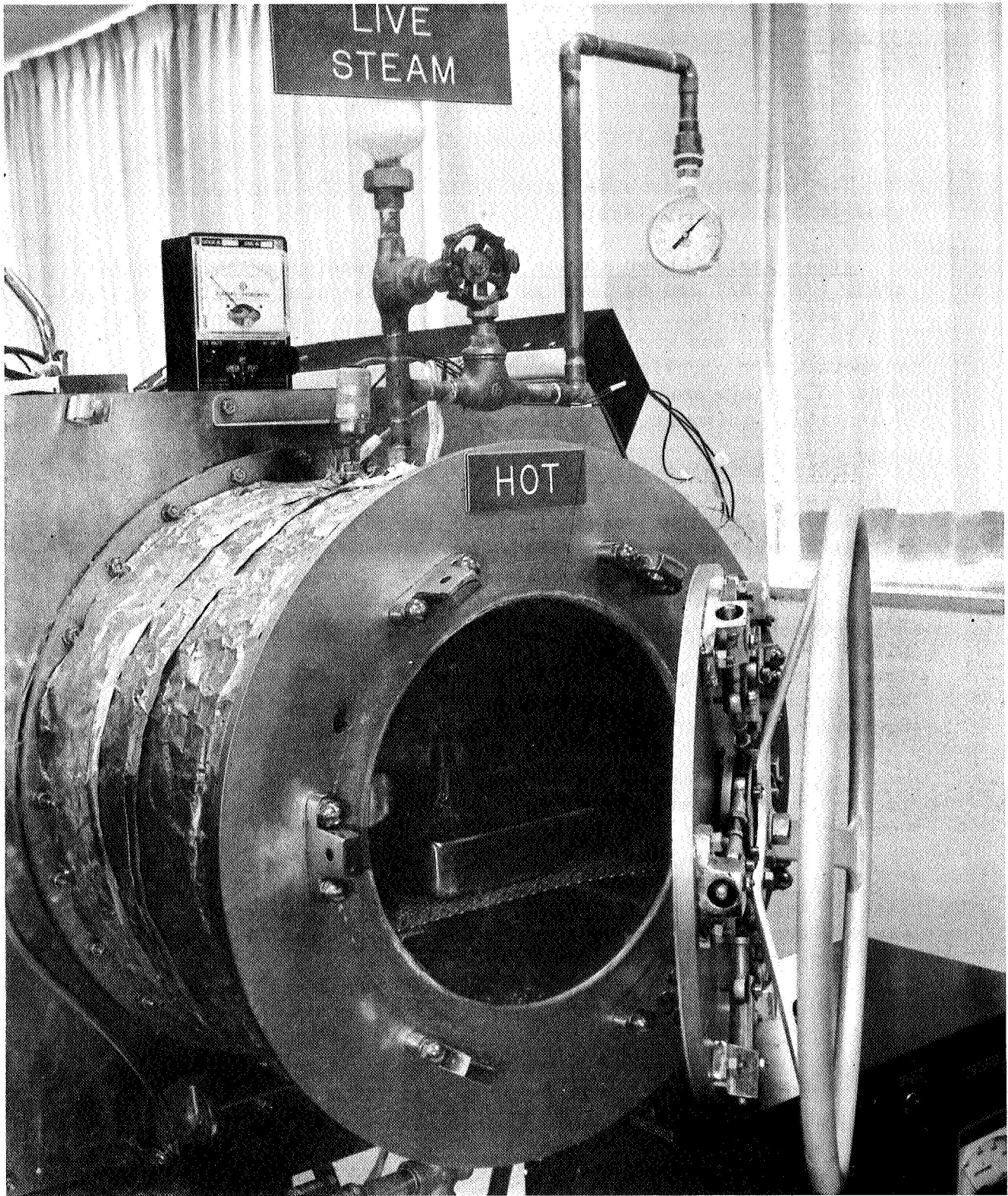


Figure A1.- Stainless Steel Sterilization Oven

APPENDIX A

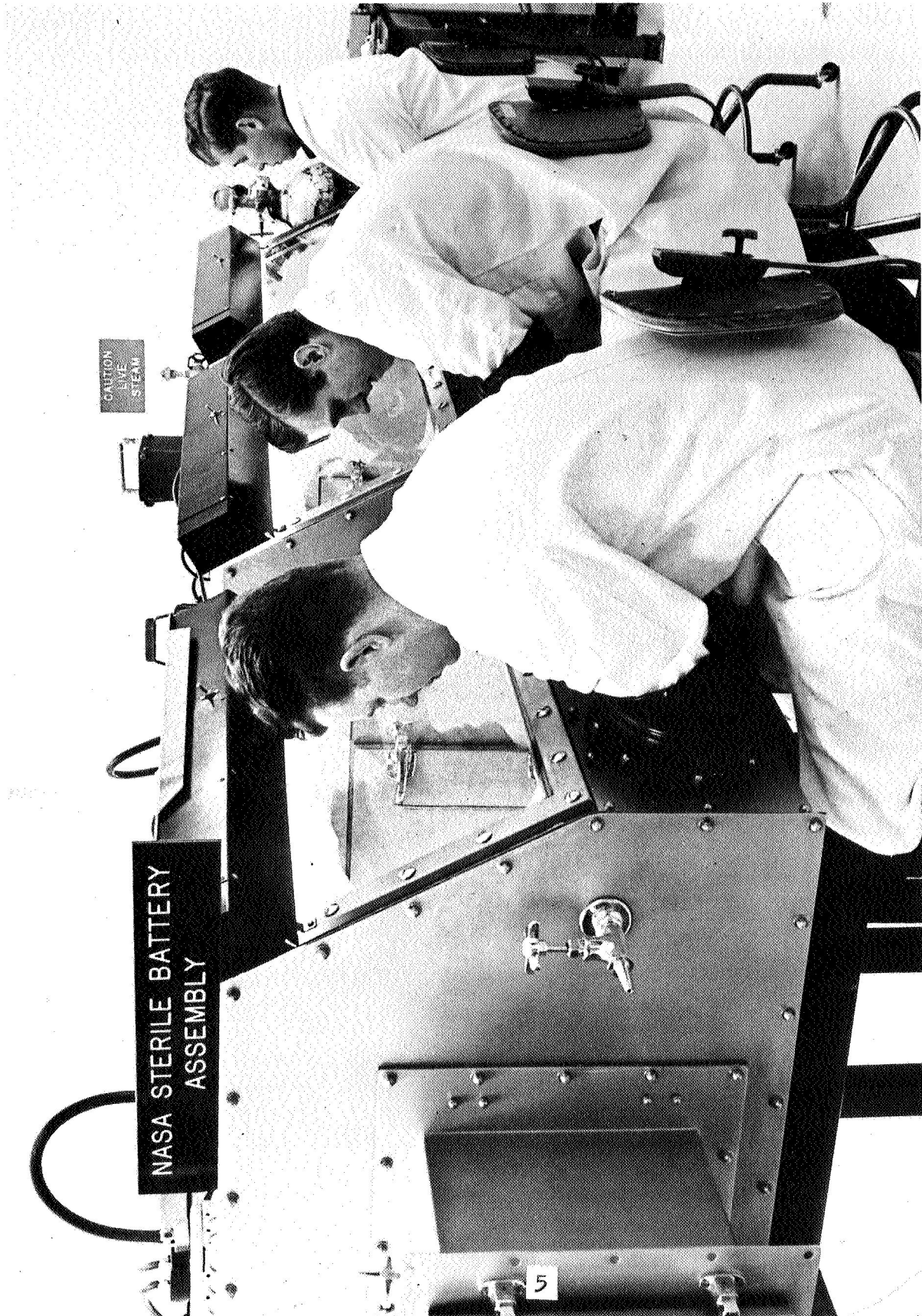


Figure A2.- Isolator Setup

## APPENDIX A

### Test Equipment

The following test equipment was used in the program:

- 1) Digital dc voltmeter - Dana model 5600;
- 2) Power supply - Capable of delivering current of 0 to 15 A within range of 0 to 50 V (Sorensen Nobatron DCR-40-TOA);
- 3) Load tester (tensile strength) - Instron model TM.

## APPENDIX B

### STERILIZATION PROCEDURES

#### Component Sterilization Tests

Test samples were placed in a vacuum oven, and the chamber was sealed. The chamber was purged with dry (99%) nitrogen. Purge was continued for 10 minutes while the chamber vent valve was adjusted for steady flow. The vent valve was then closed, and the chamber evacuated to  $70 \pm$  torr. After evacuation the chamber was backfilled with dry nitrogen. Temperature was now raised to  $135 \pm 5^\circ\text{C}$  in 6 hr followed by a nitrogen purge and maintenance at a temperature of  $135^\circ\text{C}$  for 60 hr. The oven controller was set to return to ambient temperature in approximately 6 hr, and nitrogen purge maintained after reaching ambient temperature. The nitrogen valve was closed, the chamber opened, and samples removed.

#### Cellophane Separator Sterilization Tests at 135 and $125^\circ\text{C}$

Four standard separator sheets (19.1x5.87 in.) were wrapped on a fiberglass cylinder (4-in. diameter) and covered with two layers of aluminum foil. The foil was folded over the ends of the drum and was taped in place with sterilization indicator tape. The foil package was placed in the oven and heated to 135 or  $125^\circ\text{C}$  for 60 hr in a dry nitrogen atmosphere. After termination of the heat cycle and cooldown, the foil-wrapped cylinder was removed from the oven and examined for physical and dimensional changes.

## APPENDIX C

### STERILE ASSEMBLY OPERATIONS

This appendix describes the material requirements and the procedures involved in conducting the sterile assembly operation. Sufficient detail has been incorporated to permit other investigators to follow the operations.

#### Cell Assembly Equipment and Materials

The following list of support items are needed to carry out the cell assembly procedures (see figs. C1 and C2):

- 1) Plate wrapping platform (MM-BAT7656-1) - Glass filled melamine, mounted on an aluminum base;
- 2) Positive plate folding form (MM-BAT7656-1) - Glass filled melamine plate, 1.81x8.0x0.015 in.;
- 3) Positive plate "U" fold rod (MM-BAT7656-3) - Stainless steel rod, 4.0x0.06 in. in diameter;
- 4) Cell pack assembly fixture (MM-BAT7656-4) - Phenolic sides and phenolic impregnated linen top, 1.89x3.18x1.58 in.;
- 5) Terminal height locating plate (MM-BAT7656-4B) - Fiberglass, 3.0x2.5x0.12 in.;
- 6) Soldering shield (MM-BAT7656-4D) - Fiberglass, 3.0x2.5x0.12 in.;
- 7) Carbon resistance soldering tool (MM-BAT7656-5) - 3 Vac 600 W;
- 8) Soldering tool power supply - 120 Vac primary, 3 Vac secondary, 10 A output;
- 9) Cell clamping frame (MM-BAT7656-6);
- 10) Terminal holding wrench (MM-BAT7656-4C);
- 11) Solder - SN60, Solid 1/16-in. diameter;
- 12) Kester paste solder flux;
- 13) Wire cutters;
- 14) Nut and cap wrench (MM-BAT7656-7);
- 15) Funnel, glass;
- 16) Nylon cement - Prepared by mixing 10 g Resorcinol with 10 g of 80% ethyl alcohol.



## APPENDIX C

### Legend:

- 1 Plate folding platform
- 2 Cell pack assembly fixture
- 3 Solder flux
- 4 Solder
- 5 Wire cutters
- 6 Terminal height locating plate
- 7 Terminal holding wrench
- 8 Soldering shield
- 9 Carbon resistance soldering tool

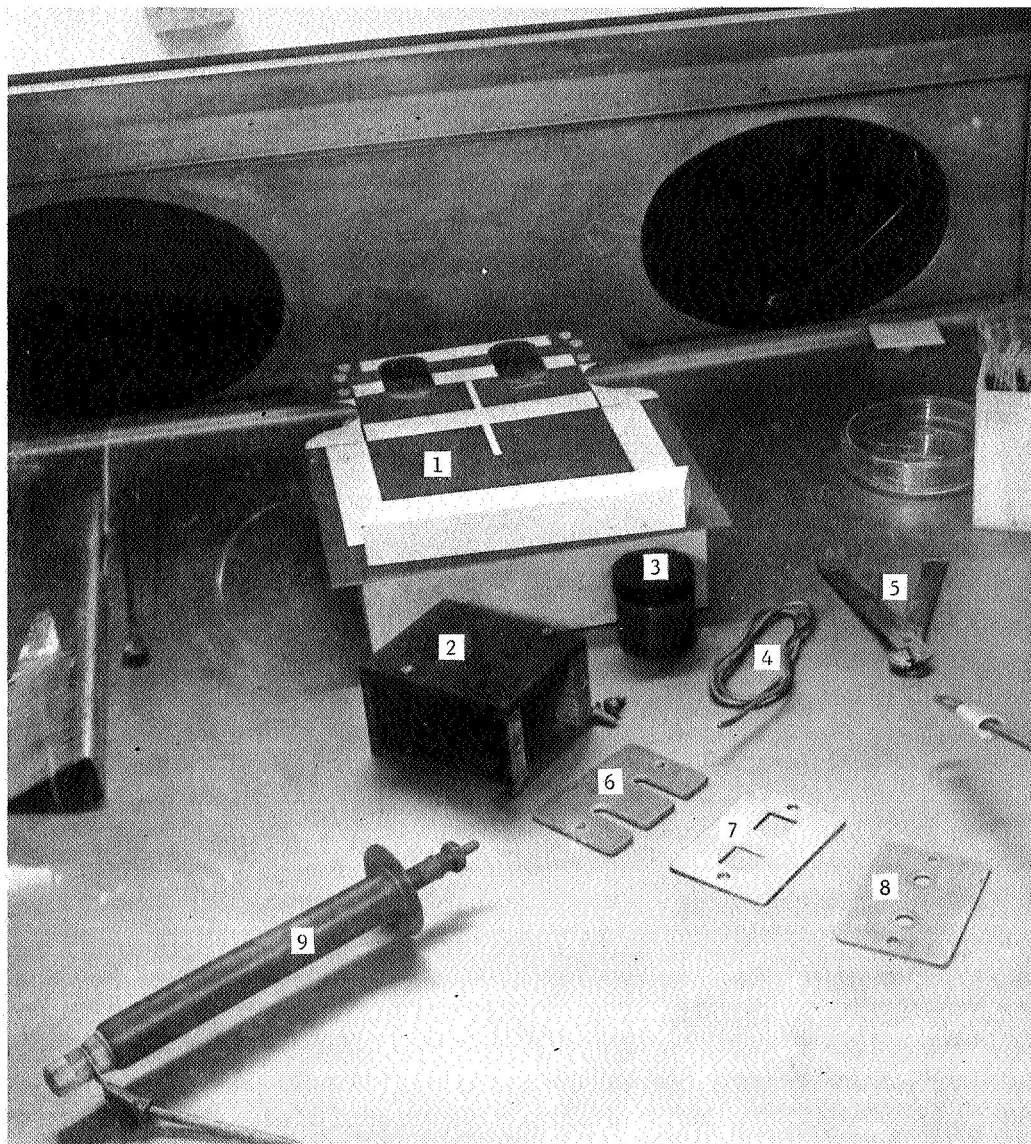


Figure C1.- Cell Assembly Equipment and Materials

APPENDIX C

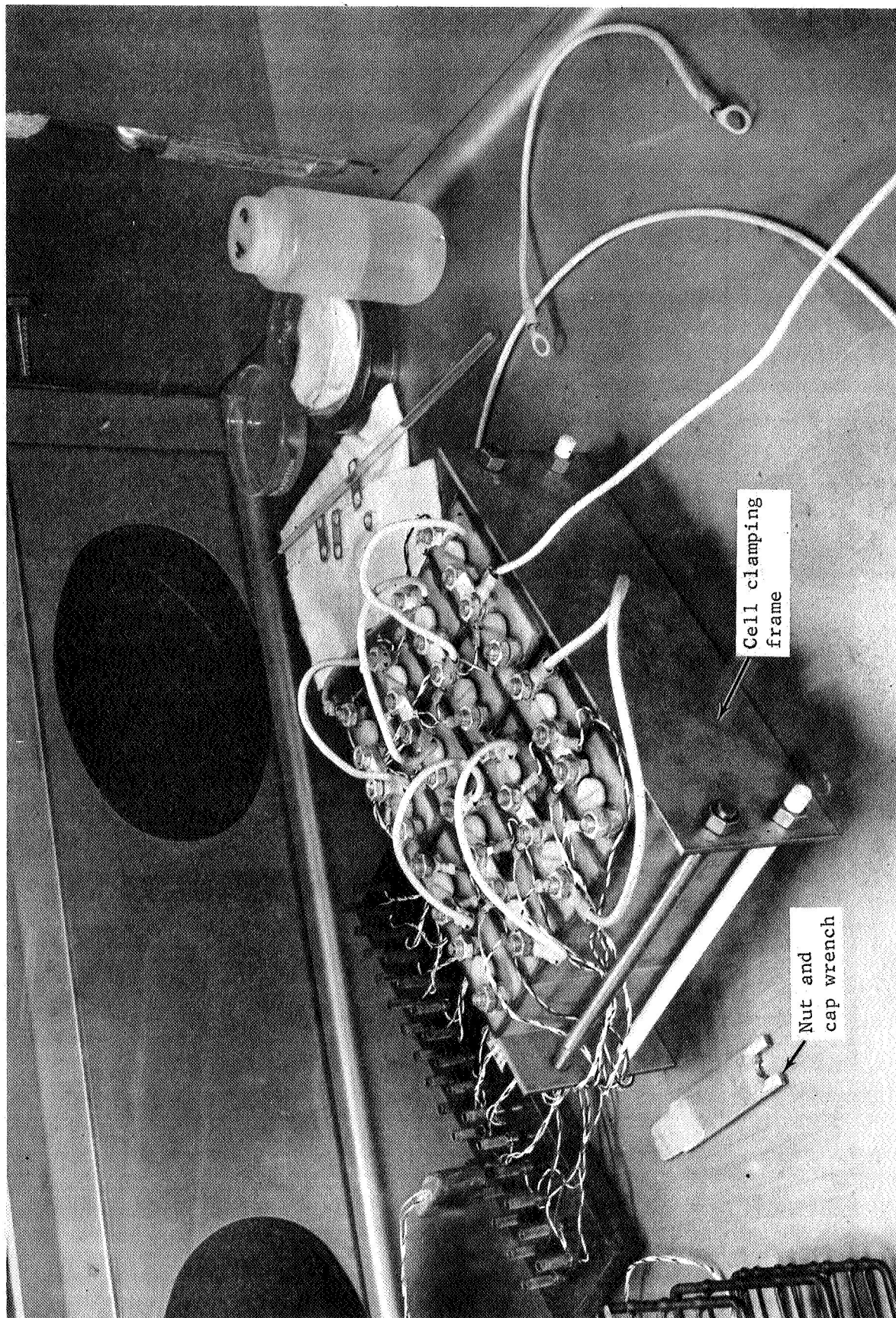


Figure C2.- Electrical Checkout

## APPENDIX C

### Cell Components

The following basic cell components are required to assemble the batteries constructed in this program (fig. C3):

- 1) Positive plate (15 required) - ESB/EMED 7-10987-1;
- 2) End negative plate (2 required) - ESB/EMED 7-10986-2;
- 3) Negative plate (14 required) - ESB/EMED 7-10986-1;
- 4) Nylon taffeta - ESB/EMED 150-10124-1N and 2N, 7 (-2N) sheets required, 1 (-1N) sheet required;
- 5) Cellophane (8 sheets required) - ESB/EMED 150-10124-1C;
- 6) Terminal post (2 required) - ESB/EMED 150-10006-6;
- 7) Cell case (1 required) - Nylon MB 57899;
- 8) Liquid vinyl - Okuns liquid vinyl;
- 9) Gasket (2 required) - ESB/EMED 150-10014-18;
- 10) Cell cover (1 required) - ESB/EMED 7-10996;
- 11) Flat washer (2 required) - ESB/EMED 150-10015-10;
- 12) Hex nut (4 required) - ESB/EMED 150-10013-2;
- 13) Vent plug (1 required) - ESB/EMED 150-10098-2;
- 14) Lock washer (2 required) - ESB/EMED 150-10041-3;
- 15) Electrolyte - Potassium hydroxide (KOH), 1.399 specific gravity, 72 ml/bottle.

### Isolator Decontamination

Before the start of sterile assembly, the interior of the isolator was decontaminated by flooding the total system with Oxyfume 12 at a relative humidity of 40% at room temperature for five days.

### Dry Heat Sterilization of Cell Components

Cell components were prepared as described below before the passclave dry heat sterilization.

Positive plates (15), negative plates (14), and negative end plates (2) required for assembly of each cell were packaged separately by wrapping the groups of plates in aluminum foil. A spore strip was placed in the packages of positive plates and negative plates.



## APPENDIX C

### Legend:

- |    |                |
|----|----------------|
| 1  | Cellophane     |
| 2  | Nylon taffeta  |
| 3  | Cell case      |
| 4  | Terminal post  |
| 5  | Hex nut        |
| 6  | Lock washer    |
| 7  | Flat washer    |
| 8  | Gasket         |
| 9  | Cell cover     |
| 10 | Electrolyte    |
| 11 | Positive plate |
| 12 | Negative plate |

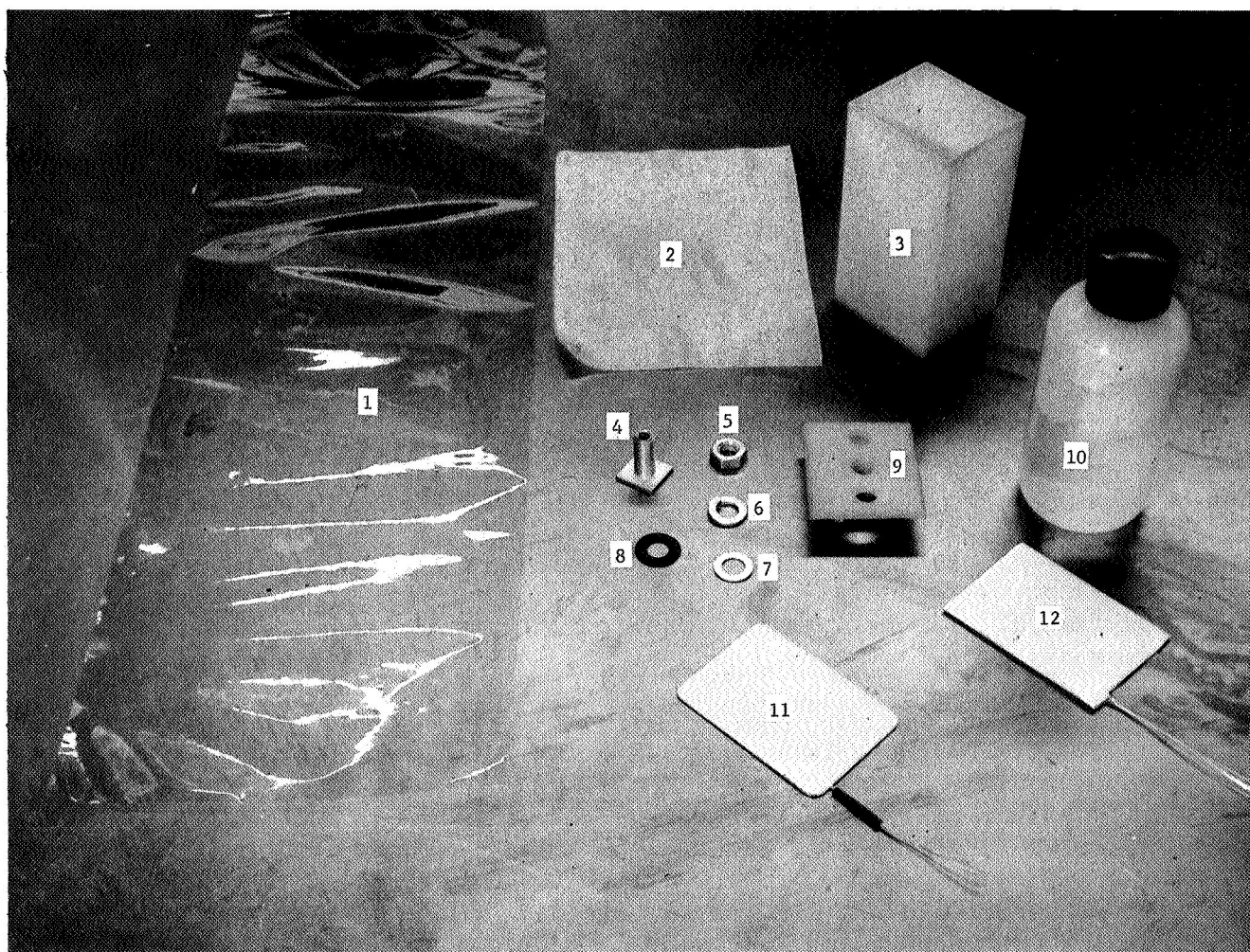


Figure C3.- Cell Components

## APPENDIX C

Terminal posts, lock washers, flat washers (2 each per cell), and hex nuts (4 per cell) were packaged in aluminum foil. A spore strip was included in these packets also.

Nylon taffeta sheets (7 large and 1 small per cell) and two spore strips were wrapped in chromatography paper. The paper was folded over the taffeta, and the edges were sealed with masking tape.

Cellophane sheets (8 per cell) were rolled in chromatography paper, and the roll was inserted into an anaerobic jar. Two spore strips were used with the cellophane. The lid was then placed on the jar and clamped in place.

Vent plugs and seals (1 each per cell) and a spore strip were packaged in an aluminum foil package.

The dry heat cycle was performed as follows:

- 1) Turn heater blanket on to preheat passclave;
- 2) Preheat approximately 4 hr;
- 3) Place parts into passclave and lock door;
- 4) Open nitrogen input and output valves to passclave and purge for 5 minutes at 2 lb pressure;
- 5) Shut off nitrogen input and output valves to passclave;
- 6) Check temperature for 135°C and maintain for 60 hr;
- 7) Turn off heater;
- 8) Open nitrogen input and output valves to passclave;
- 9) Set nitrogen regulator to approximately 2 lb;
- 10) Cool passclave to approximately 40°C;
- 11) Turn off nitrogen input and output valves on passclave;
- 12) Open the inner door and remove parts.

### Moist Heat Sterilization Cycle

Equipment and materials subjected to moist heat sterilization were:

- 1) Plate folding platform;
- 2) Positive plate wrapping form;

## APPENDIX C

- 3) Positive plate U-fold rod;
- 4) Cell pack assembly fixture;
- 5) Terminal height locating plate;
- 6) Lug holding wrench;
- 7) Soldering shield;
- 8) Cell holding frame;
- 9) Solder;
- 10) Funnel;
- 11) Nylon cement, in test tube;
- 12) Cell cases and covers (after dry cycle to rejuvenate);
- 13) Torque wrench;
- 14) Wire cutters.

The moist heat sterilization procedure for cell materials and equipment is as follows:

- 1) Turn on steam generator;
- 2) Open valves on top and bottom of autoclave and purge with steam for 5 minutes before putting in parts;
- 3) Shut off steam input valve;
- 4) As soon as steam pressure is zero in autoclave, open the door and place parts inside;
- 5) Close door and turn on steam input valve with drain valve open;
- 6) Purge chamber with live steam for 5 minutes;
- 7) Close drain valve;
- 8) Autoclave for 30 minutes;
- 9) Shut off steam input valve;
- 10) Decrease steam pressure by slowly opening drain valve until pressure is zero;
- 11) Close drain valve;
- 12) Open nitrogen input valve to autoclave and pressurize line to approximately 2 lb;
- 13) Open drain valve to drain nitrogen and accumulated moisture;

## APPENDIX C

- 14) Cool autoclave with nitrogen until temperature is 40°C;
- 15) Shut off nitrogen input and drain valves;
- 16) Open inner door and remove parts.

### Equipment and Material Placement in Isolators

All equipment and materials were transferred directly from the passclave into the first isolator. All assembly materials and equipment were not transferred to the second isolator until biological security was verified. Isolator 3 was used for the filling of the sterile cells with electrolyte and for electrical testing.

Note: All passway doors were closed before opening the passclave or a passway door to an adjoining isolator. All sterile items were passed through the isolator system as follows:

- 1) Equipment and materials were transferred into the passway between the first and second isolators by opening the passway door in the first isolator, placing the equipment and materials in the passway, and closing the door;
- 2) The passway door in the second isolator was then opened, the materials and equipment were removed, and the door was closed;
- 3) Passage of equipment and materials into the third isolator was accomplished in the same manner.

### Isolator Environment

Before the start and throughout the assembly operations, relative humidity of 40% was maintained inside the isolators to assist in the humidification of the nylon cell elements and the cellophane separators. In addition, a positive pressure (1 in. water) was maintained inside the isolator system at all times. All germicidal lamps in the isolator system were turned on at the start of the program. The lamps were not turned off at any time during the program.

## APPENDIX C

### Step-by-Step Cell Assembly Operation

Positive plate wrap. - Place one sheet of cellophane (ESB/EMED 150-10124-1C), one sheet of nylon taffeta (ESB/EMED 150-10124-2N), the positive plate wrapping form (MM-BAT7656-1), and two positive plates (ESB/EMED 7-10987-2) on the plate folding platform (MM-BAT-7656-2). Fold cellophane and taffeta over positive plates. Grip the plates and wrapping form securely, fold over to obtain one complete layer of taffeta around the plates and five layers of cellophane around the taffeta as shown in figure C4.

Note: Tension is applied to the cellophane to ensure a tight fold.

Partially withdraw the wrapping form past the space between the bottom of the plates and place the U-fold rod (MM-BAT7656-3) in the center of this space as shown in figure C5. Fold the assembly so the top edges of the plates and the top edges of the separator material are aligned, respectively. Withdraw the wrapping form and U-fold rod.

Cell pack assembly. - Place cell pack assembly fixture (MM-BAT7656-4) in position for stacking plates. Place one end-negative plate (ESB/EMED 7-10986-4) in the assembly fixture. Insert one negative plate (ESB/EMED 7-10986-1) in the U-fold between the positive plates of the positive plate U-fold packet, so the negative plate top edge is aligned with the positive plate top edges as shown in figure C6.

Note: Negative plate lead is to the opposite edge of the packet from the positive plate leads.

Place the three-plate U-fold packet (two positive and one negative) in the pack assembly fixture on top of the negative end plate. Repeat the positive plate wrap steps until 14 positive plates and 13 negative plates have been placed in the assembly fixture. The 15th positive plate is wrapped with a small piece of taffeta (ESB/EMED 150-10124-1NO) and has no positive plate in one side of the U-fold pack.

Note: Care should be taken to ensure all plates are oriented so the negative leads are to one edge of the pack and positive leads to the other edge.

Insert one end negative plate (ESB/EMED 7-10986-2) in the U-fold of the 15th positive plate packet. The empty section of the packet should be on top when placed in the assembly fixture. Install

APPENDIX C



Figure C4.- Wrapping Positive Plate



APPENDIX C

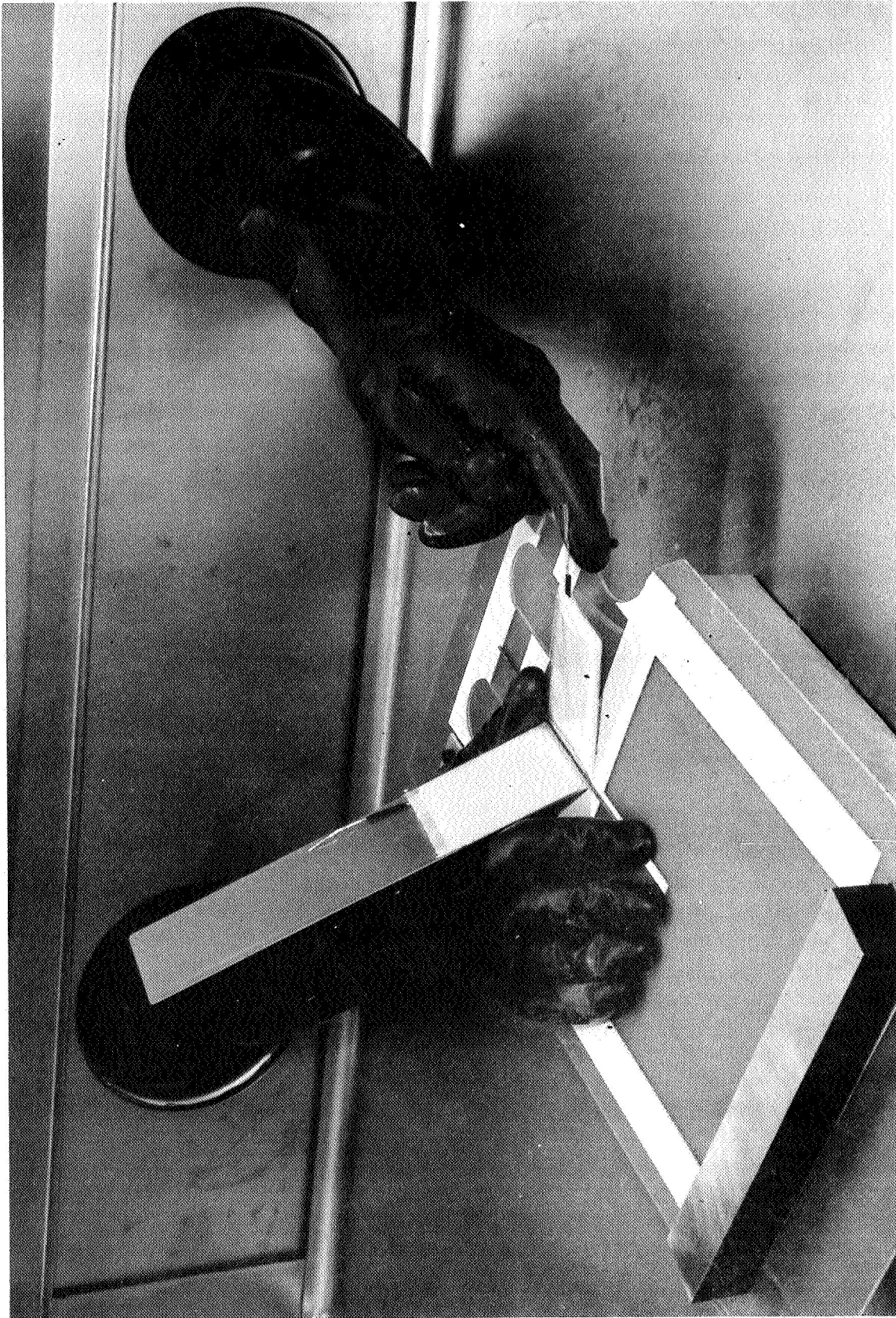


Figure C5.- Positive Plate U-Fold

## APPENDIX C

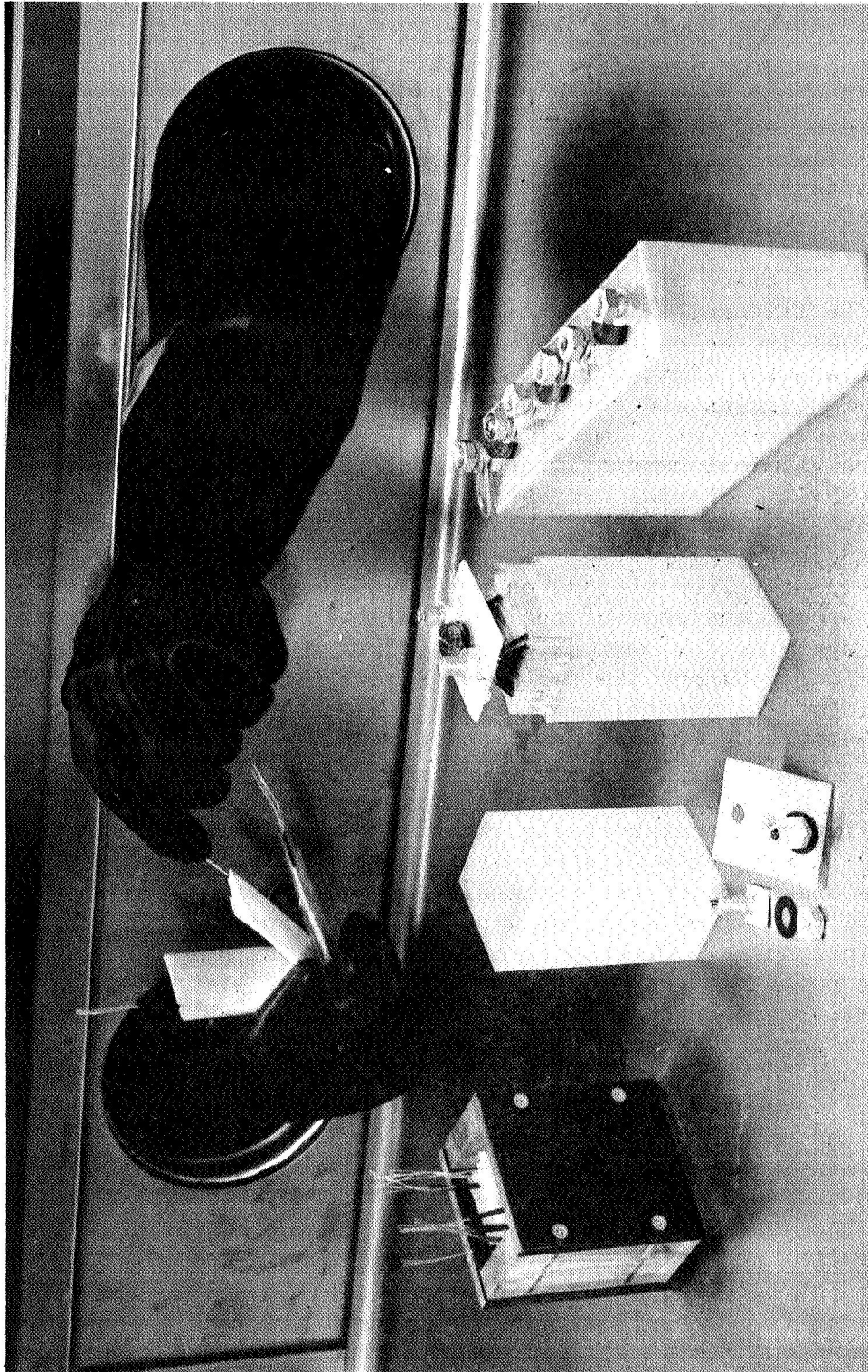


Figure C6.- Inserting Negative Plate



## APPENDIX C

the assembly fixture front panel (MM-BAT7656-4A) and place fixture on end. Inspect and adjust insulation tubing to touch top of respective plates. Inspect and adjust plates so the top edges are aligned and the positive and negative plate leads are in straight rows. Form lead wires, above top of separators toward the center plates. Place terminal height locator plate (MM-BAT7656-4B) on top of assembly. Insert terminal lead wires in terminal post (ESB/EMED 150-10006-6) so terminals rest on height locator plate. Raise terminal post high enough to permit bundling of lead wires with a small piece of silver wire. Remove the terminal posts, invert the fixture, and cut the lead wire bundles so they will be  $0.19 \pm 0.03$  in. below the top of the terminal post when reassembled. Place the fixture back in the upright position. Replace the terminal posts and remove the tie wires from the lead bundles. Inspect assembly for terminal post position and plate alignment. Place the terminal holding wrench (MM-BAT7656-4C) and soldering shield (MM-BAT7656-4D0) over terminal posts and secure in position with two  $\frac{1}{2}$ -in. 10-32 screws. Apply a small amount of flux to the end of the terminal posts. Solder the lead wires in the terminal posts by placing the tip of the resistance soldering tool (MM-BAT-7656-5) at the top edge of the terminal post as shown in figure C7, and apply solder until approximately  $\frac{3}{8}$  in. has flowed into the terminal. Remove the two 10-32 screws, the soldering shield, terminal holding wrench, and height locator plate. Inspect for solder below the terminal posts and on top of the cell pack.

Final cell assembly. - Remove cell pack assembly from the assembly fixtures, and carefully insert in a cell case (ESB/EMED MB-57899) until the separators are even with the case top edge. Coat the underside of the terminals and all exposed wire with liquid vinyl. Allow 2 hr for vinyl to dry. Install nylon cover on terminal posts. Check polarity to assure the positive and negative posts are inserted in holes identified with plus (+) and minus (-), respectively. Place flat washer (ESB/EMED 150-10015) over each terminal post. Secure cover to terminal posts using one hex nut (ESB/EMED 150-10013) on each terminal. Insert the cell pack completely in the case so the cover is firmly in position in the case shoulder. Cement the cover to the case using nylon cement, and place cell in clamping frame (MM-BAT7656-6). Allow 48 hr for cement to dry before filling with electrolyte.

Cell filling. - The cells were filled by placing a glass funnel into the filter opening in the lid (see fig. C8). The cap was removed from the electrolyte storage bottle, and the contents of the bottle (72 ml) were poured into the cell. The funnel was removed, and the vented plug was placed in the filler opening. The plug was tightened until the gasket was just compressed.



Figure C7.- Soldering



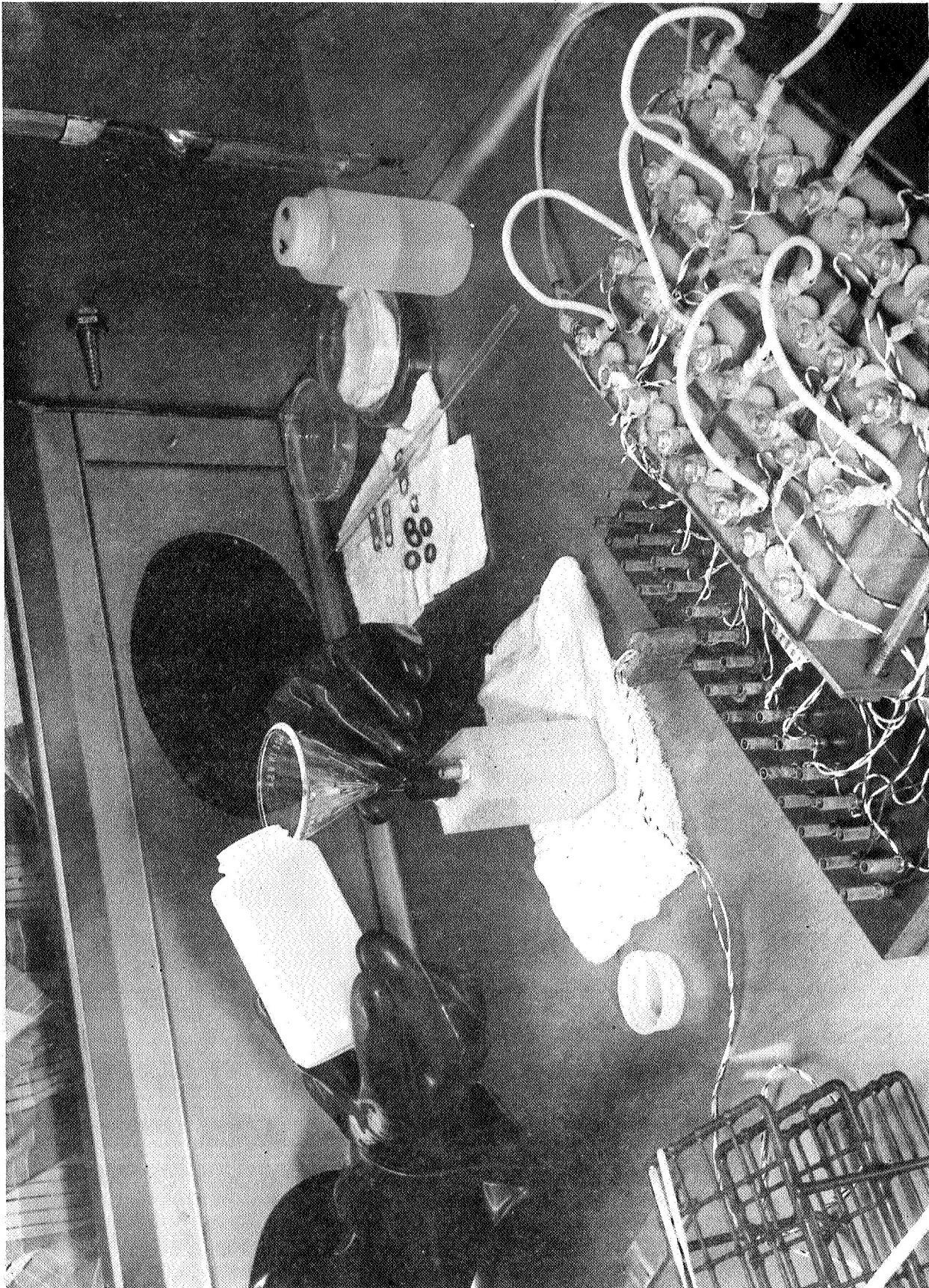


Figure C8.- Filling Cell

## APPENDIX C

Cells were kept in an upright position and allowed to stand for 3 to 7 days before beginning electrical testing.

## APPENDIX D

### CHARGE/DISCHARGE TEST PROCEDURE

#### Formation Cycle

Charge sequence. - The charge sequence is as follows:

- 1) Connect test cells to the charge circuit as shown in figure D1;
- 2) Read and record the open circuit voltage of each cell;
- 3) Record test start time;
- 4) Adjust power supply to obtain a 2-A charge reading on the ammeter;
- 5) Check millivolt reading across the shunt and adjust power supply current to obtain a 20-mV reading on the digital voltmeter;
- 6) Read and record voltage and current of each cell every half hour during charge;
- 7) Check millivolt reading across shunt every hour to verify charge current accuracy;
- 8) Discontinue charge of each cell as its respective voltage reaches 2.1 V.

Letdown discharge. - The letdown discharge sequence is as follows:

- 1) Adjust the variable resistance of figure D2 to the proper value for the number of test cells being discharged. Cells other than test specimens (Ag-Zn type) are used for calibration purposes. The resistance value then remains constant for the test duration;
- 2) Remove calibration cells and connect test cells to the charge circuit as shown in figure D2;
- 3) Read and record the open circuit voltage of each cell;
- 4) Record test start time;
- 5) Adjust the power supply to obtain a 4-A discharge current reading on the ammeter;
- 6) Check the millivolt reading across the shunt, and adjust power supply current to obtain a 20-mV reading on the digital voltmeter;

# APPENDIX D

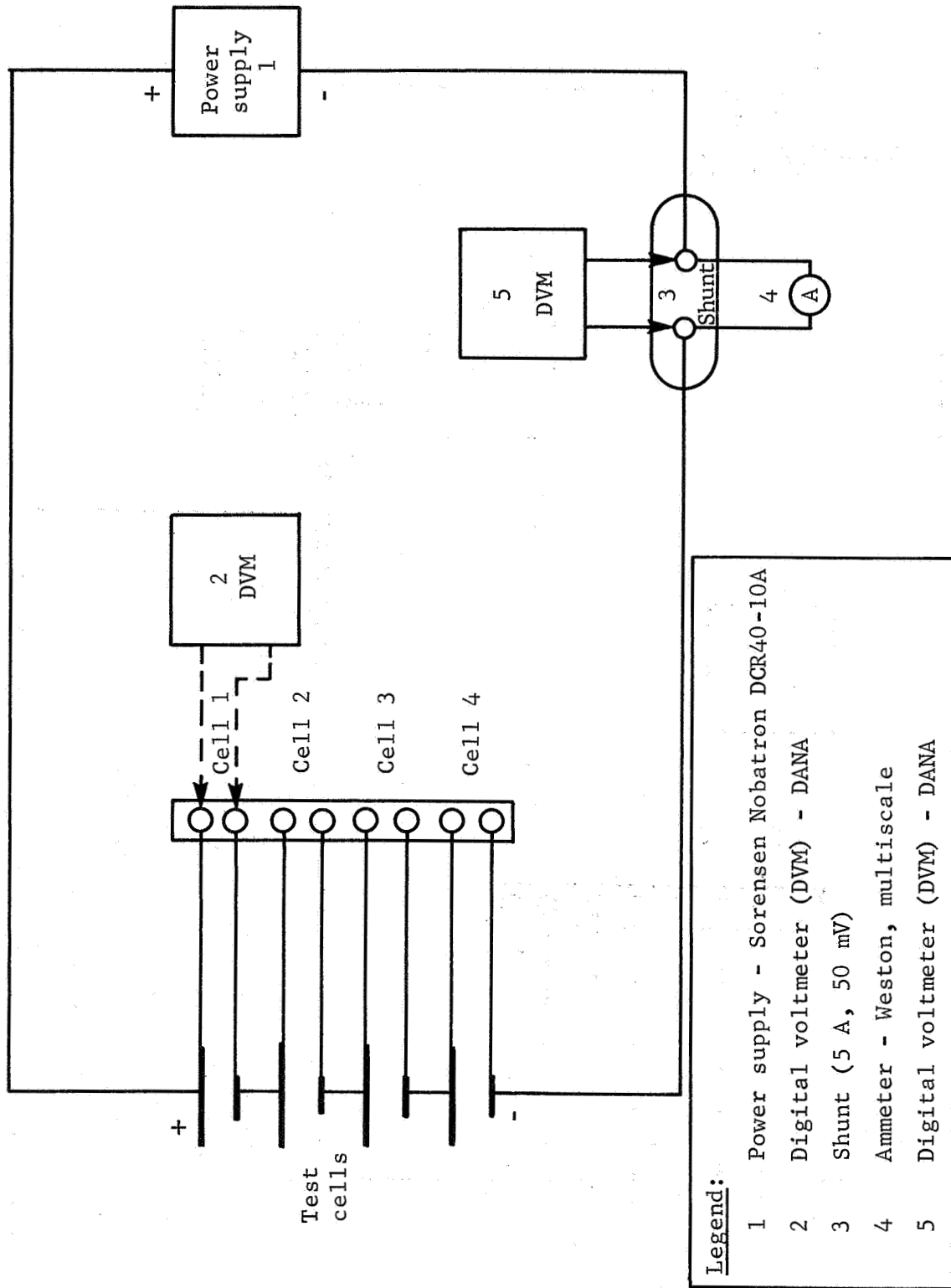


Figure D1.- Charging Circuit Diagram

# APPENDIX D

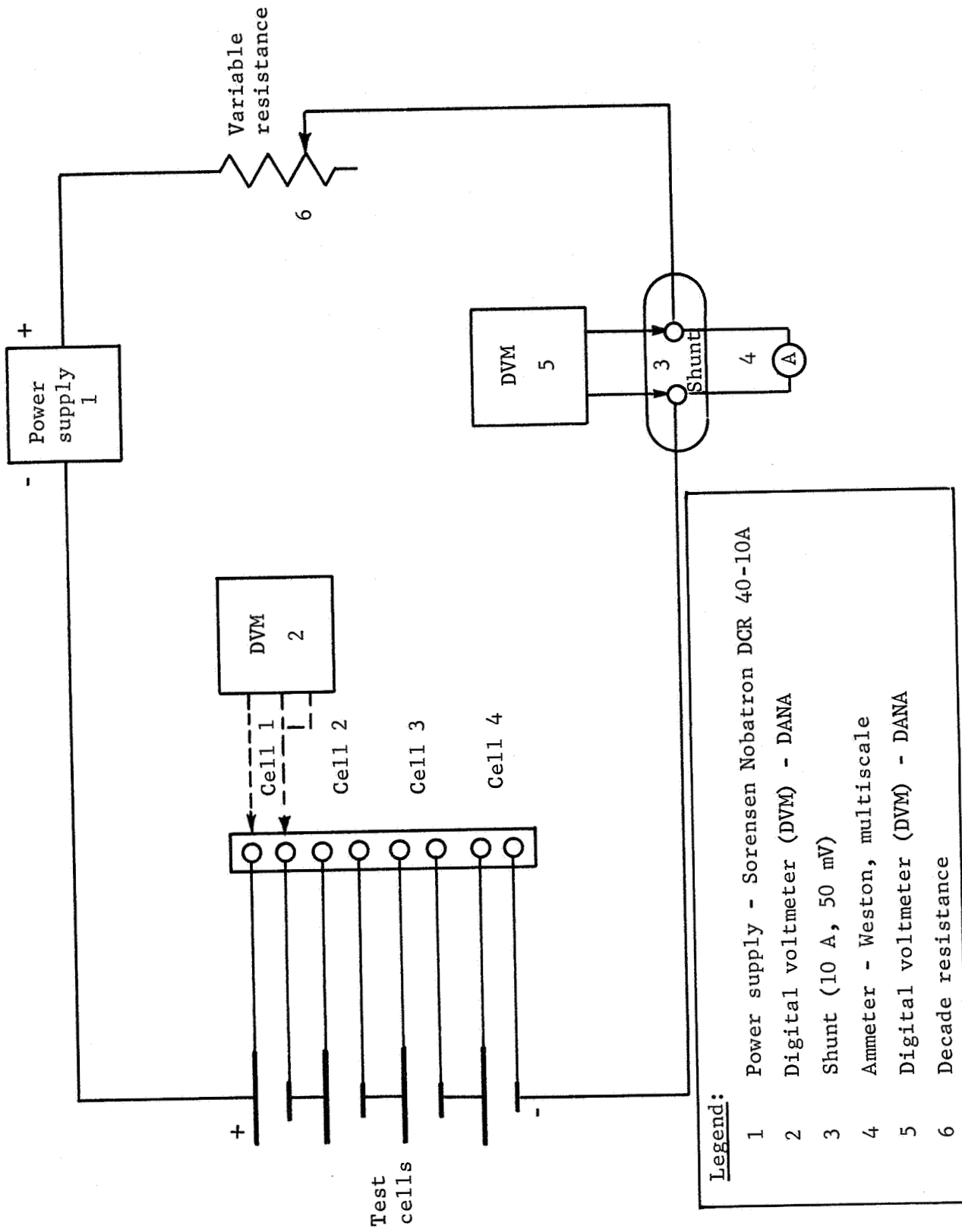


Figure D2.- Discharging Circuit Diagram

## APPENDIX D

- 7) Read and record the voltage and current of each cell every half hour during discharge;
- 8) Check the millivolt reading across the shunt every hour to verify discharge current accuracy;
- 9) Discontinue discharge of each cell when its respective voltage reaches 1.00 V.

### Operational Cycle

Charge sequence. - The charge sequence for the operational cycle is an identical procedure as that given above for the formation cycle.

Discharge sequence. - The discharge sequence is as follows:

- 1) Adjust the variable resistance of figure D2 to proper value for the number of cells being discharged. Cells other than test specimens (Ag-Zn type) are used for calibration purposes. The resistance value then remains constant for the test duration;
- 2) Remove calibration cells and connect test cells to the charge circuit as shown in figure D1;
- 3) Read and record the open circuit voltage of each cell;
- 4) Record test start time;
- 5) Adjust the power supply to obtain a 10-A discharge current reading on the ammeter;
- 6) Check the millivolt reading across the shunt, and adjust power supply current to obtain a 50-mV reading;
- 7) Read and record the voltage and current of each cell every half hour during discharge;
- 8) Check the millivolt reading across the shunt every hour to verify discharge current accuracy;
- 9) Discontinue discharge of each cell when its respective voltage reaches 1.00 V.



## APPENDIX E

### MICROBIOLOGICAL CONTROL AND ASSAY PROCEDURES

#### Media Preparation

Peptone water. - Peptone water (Difco) was prepared by dissolving 10 of the powder in a liter of distilled water. The resulting solution was then dispensed into plastic bottles (8-oz polypropylene bottles) and autoclaved (121°C, 20 minutes). The bottles were then incubated for 5 days to check for sterility.

Trypticase Soy Broth (TSB). - Trypticase Soy Broth (BBL) was prepared by adding 30 g of the powder to a liter of distilled water. The resulting solution was then dispensed into tube or bottle and autoclaved (121°C, 20 minutes). The solutions were then incubated for 5 days to check for contamination. If no visible contamination was noted, the solutions were deemed sterile and ready for use.

Trypticase Soy Agar (TSA). - Trypticase Soy Agar (BBL) was prepared by dissolving 40 g of the powder in a liter of distilled water. The resulting solution was autoclaved (121°C, 20 minutes), after which it was used for pour or streak plates. The uninoculated streak plates were incubated for 2 days to check for contamination. Pour plates were checked by making two uninoculated pour plates along with each inoculated set to be examined.

#### Sterilization Process Evaluation

Autoclave. - Items to be steam sterilized were placed in the autoclave along with a spore strip (AMSCO Corp.). The spore strip was generally located in the center of a packet of battery plates, cotton pads, or taped to the exterior of a tool, etc. In all cases, the spore strips were located in the most invulnerable position possible with regard to steam penetration. The autoclave was closed and purged for 5 minutes with live steam, after which the exhaust valve was closed. The chamber was then pressurized to approximately 21 lb and 121°C for 30 minutes. After completion of the autoclave cycle the chamber was cooled, and the parts were removed and placed in chamber 1 of the isolator system. The spore strips were removed, placed in Trypticase Soy Broth and incubated for 7 days. If no growth was noted from any of the spore strips, the parts and materials were transferred from chamber 1 to chamber 2 where actual construction took place.

## APPENDIX E

Dry heat sterilization. Before a sterilization cycle, the chamber and the parts to be sterilized were preheated to 125°C (approximately 4 hr). The parts to be sterilized were wrapped in aluminum foil along with a spore strip (one spore strip per packet) and placed in the chamber in a thick walled aluminum box. The parts were heated at 125°C for 60 hr after which they were cooled and stored in isolator 1. The spore strips were removed, placed in Trypticase Soy Broth and incubated for 7 days. If no growth was noted from any of the strips, the parts were transferred to chamber 2 for assembly.

Assembly isolators. - Prior to installation of the end panels interconnecting the three isolators, each unit was washed with fresh 2.0% peracetic acid (City Chemical Corp., New York), rinsed with sterile distilled water, and wiped dry with sterile cheese cloth. The end panels were then installed and sealed. For bio-assay spore strips were placed in each isolator and the connecting passthroughs. The isolator system was then evacuated to 12 in. of water, as measured by a water column, and backfilled with Oxyfume 12 (Union Carbide Corp., New York) until one in. of positive pressure was achieved within the system as indicated by the water column. The system was purged for an hour with dry sterile nitrogen and evacuated again to 12 in. of water and backfilled with Oxyfume 12 to a positive internal pressure of 2 in. of water. Humidity was introduced into the isolator atmosphere by evaporation of sterile water from two 100 x 15 mm petri dishes. The Oxyfume 12 was maintained in the system for 68 hours. At the end of the 68 hour period the exposed spore strips were assayed in TSB. When no evidence of growth was observed after 48 hours, the isolator system was flushed three times by alternate filling and evacuation of dry sterile nitrogen. The third filling of nitrogen was maintained as the sterile atmosphere for assembly and test of the cells. All assays were checked for growth for seven days.

### Microbiological Assay Procedures

Spore strip assays. - Spore strips to be assayed were removed from their packets in a laminar flow bench and aseptically transferred to 15 ml of sterile TSB in screw cap tubes. The tubes were then incubated for 7 days, after which each tube was checked visually for growth. A positive control and a TSB blank were used for each set of spore strips.

## APPENDIX E

Assay of isolator assembled cells. - Twelve cells, three batteries, assembled under sterile conditions were assayed after electrical testing. One cell packet consisting of cellophane, taffeta, three negative plates, two positive plates and the external hardware (terminals, including nuts and washers) was assayed from each cell (fig. E1). The components of the selected packet were placed in separate sterile bottles containing sterile peptone water (50 ml). The peptone water (1%) was autoclaved twice, once after initial preparation and again when it was passed into the sterile isolator system. The disassembly of the cells and concurrent immersion in peptone water was carried out inside chamber 1 of the isolator system. Individual bottles, of peptone water (containing a cell part) were removed from the isolator chamber and insonated for 5 minutes. After ultrasonication, the particulate matter was allowed to settle for 1 hr, after which a 10 ml aliquot was removed from each bottle and transferred to 200 ml of sterile TSB. The bottles of TSB were then incubated for 7 days. In addition, a 2 ml aliquot was pour-plated in TSA from each bottle (pour plates were made after 5 days of incubation). If no growth was noted after 7 days, one-fourth of one spore strip was added to each negative TSB bottle to check for growth inhibition.

In addition to the assay of the cell packets, the exterior of 16 of the sterile assembled cells were assayed by swab. Cotton swabs (prepared in the same manner as described below) wetted with sterile water were rubbed over the exterior of the cell case of each cell sampled. The swab was then placed in 15 ml of sterile TSB and incubated for 7 days to check for growth. A positive growth tube and a blank TSB tube were used as controls.

Assay of isolator system. - Four open petri dishes containing TSA were placed in each of the three isolators. One open petri dish was placed in each of the two connecting chambers between the isolators. Once a week the finger tips of the gloves used in the isolators were pressed onto the agar surface of the petri dishes. The petri dishes were then removed, incubated for 7 days at 32°C, and examined visually for contamination.

# APPENDIX E

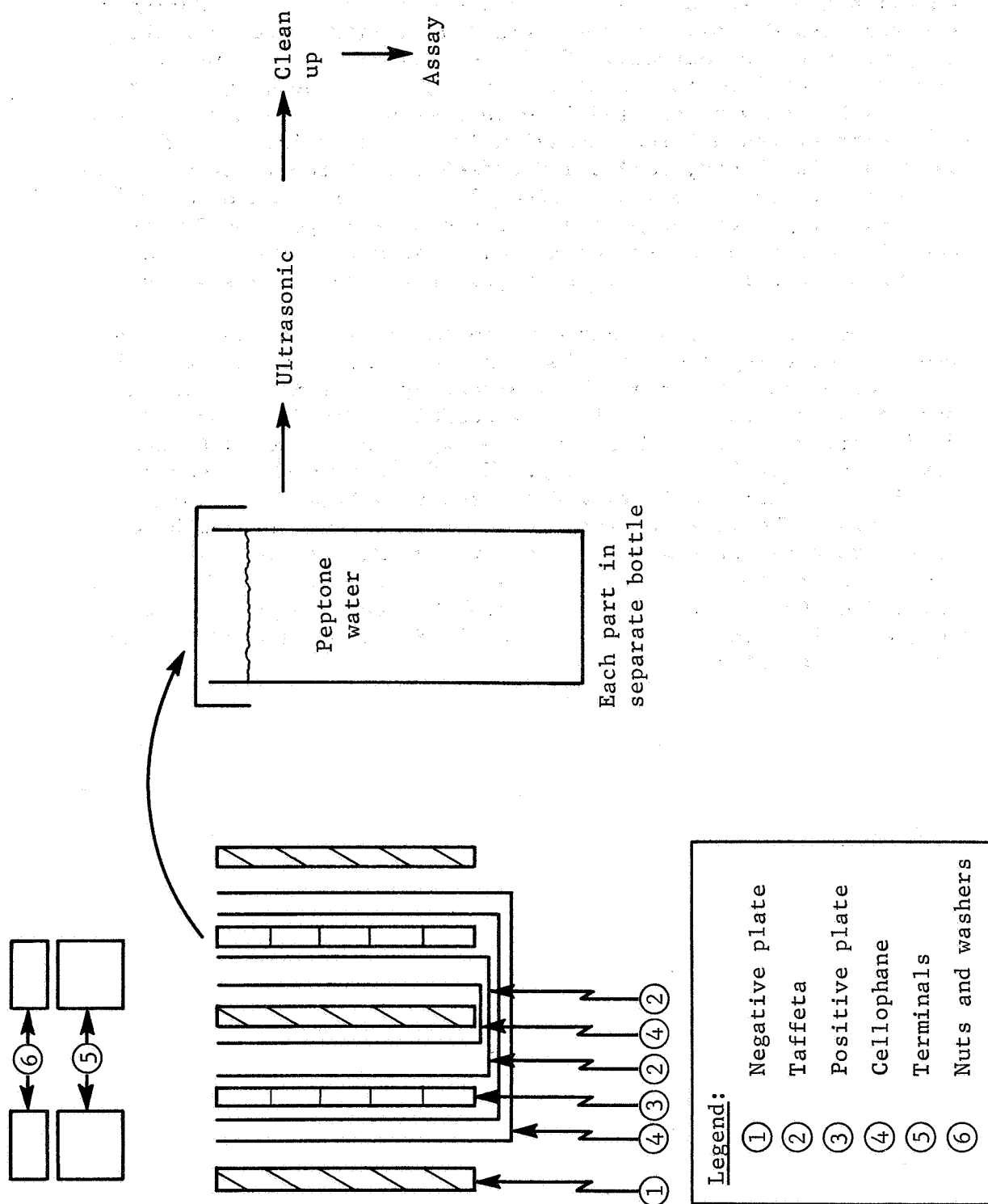


Figure E1.- Bioassay of Sterile Assembled Cells

## APPENDIX E

Subsequent to completion of the post-electrical test biological assays of the cells, the isolator system was assayed for microbial contamination. Two series of 14 swabs each were tested during a two-week period. All assay materials were introduced into the system through the autoclave following standard operating procedures. Four 4-sq-in. areas per isolator and one 4-sq-in. area per passthrough were sampled in each test series. After use, the swabs were broken off into individual tubes containing 15 ml of Trypticase Soy Broth, sealed and passed out of the isolator system. The tubes evidenced no growth after 7 days of incubation at 32°C. To check for growth inhibition, one-tenth of a spore strip was added to each tube and incubation was continued for 48 hr. At the end of this interval, profuse growth was noted in all tubes.

To check for possible airborne contamination inside the isolator system a series of millipore filters was connected to all the inlet/outlet valves of the isolator system (fig. 14, which appears in the section entitled BIOLOGICAL ASPECTS). The filter (0.45  $\mu$  field monitors, Millipore Corp.) were connected to the valves with sterile Tygon tubing. The filters were checked by removing the field monitors and transferring them to a laminar flow bench. The monitors were opened in the laminar flow bench, and the test filter was picked out with sterile forceps and dropped into a plastic bottle containing 50 ml of sterile TSB. The bottles containing the filters were then incubated for 7 days. In addition, a 2-ml aliquot was pour-plated in TSA from each bottle after 5 days of incubation.

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